10/731,733

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NEWS	14	DEC 30	EPFULL: New patent full text database to be available on STN
NEWS	15	DEC 30	CAPLUS - PATENT COVERAGE EXPANDED
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NEWS EXPRESS	JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005
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=> s 1738-25-6/rn
           305 1738-25-6
             5 1738-25-6D
L1         302 1738-25-6/RN
                 (1738-25-6 (NOTL) 1738-25-6D )

=> s 109-55-7/rn
          3694 109-55-7
          1103 109-55-7D
L2        2673 109-55-7/RN
                 (109-55-7 (NOTL) 109-55-7D )

=> s l1 and l2
L3          44 L1 AND L2

=> s ni or nickel
        580021 NI
          3698 NIS
        582255 NI
                 (NI OR NIS)
        567492 NICKEL
           193 NICKELS
        567520 NICKEL
                 (NICKEL OR NICKELS)
L4      787174 NI OR NICKEL

=> s l3 and l4
L5          15 L3 AND L4

=> d l5 1-15 abs ibib
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L5 ANSWER 1 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB A low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising sponge nickel and aqueous alkali is described.

ACCESSION NUMBER: 2004:609968 CAPLUS
DOCUMENT NUMBER: 141:140075
TITLE: Low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising sponge nickel and aqueous alkali
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 8 pp., Cont.-in-part of U.S. Ser. No. 327,765.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004147784	A1	20040729	US 2003-731733	20031209
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A2 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 141:140075

L5 ANSWER 2 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB A low-pressure hydrogenation process for the production of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile (I) comprises: feeding hydrogen and 3-(dimethylamino)propionitrile into a low-pressure reactor containing a sponge nickel catalyst, at least one Group IA alkali metal hydroxide (e.g., potassium hydroxide), and water to form a reaction medium; heating the reaction medium to 70-100°; pressurizing the reactor to 45-500 psig; and hydrogenating the nitrile to form I.

ACCESSION NUMBER: 2004:589527 CAPLUS
DOCUMENT NUMBER: 141:123405
TITLE: Low-pressure catalytic hydrogenation process for the manufacture of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: PCT Int. Appl., 20 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 6660887	B1	20031209	US 2002-327765	20021223
US 2004147784	A1	20040729	US 2003-731733	20031209

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 141:123405

L5 ANSWER 3 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB A process for the production of 3-(dimethylamino)propylamine (I) in high (>99%) purity from 3-(dimethylamino)propionitrile utilizing a low-pressure hydrogenation process is described which comprises contacting the nitrile with hydrogen at low pressure in the presence of a sponge nickel catalyst and ≥1 Goup IA metal hydroxide at 70-100°/45-150 psig. The improvement in the process resides in a combination of carrying out the hydrogenation process at low pressures and temps. in the presence of a catalytic amount of caustic base in order to give a I selectivity of >99.60%.

ACCESSION NUMBER: 2003:961180 CAPLUS
DOCUMENT NUMBER: 140:17730
TITLE: Low-pressure hydrogenation process and catalyst system for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: U.S., 7 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060039	A2	20040722	WO 2003-US29721	20030919
WO 2004060039	A3	20040826		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 2004147784	A1	20040729	US 2003-731733	20031209
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209

L5 ANSWER 3 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

OTHER SOURCE(S): CASREACT 140:17730
REFERENCE COUNT: 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 4 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB Primary amines were prepared by hydrogenation of nitriles in the presence of catalysts containing Co and optionally Ni as well as ≥1 doping metal on a particulate substrate, whereby the Co and optional Ni have an avg. particle size of 3-30 nM. Thus, dimethylaminopropionitrile was hydrogenated in the presence of a suspension catalyst [prepared from Co(NO3)2, Ni(NO3)2, and Y(NO3)3 and aluminosilicate powder] at 80° in the presence of NH3 and 80 bar H2 to give dimethylaminopropylamine in 98.4% selectivity.

ACCESSION NUMBER: 2003:332011 CAPLUS
DOCUMENT NUMBER: 138:337704
TITLE: Preparation of primary amines via reduction of nitriles in the presence of supported cobalt catalysts containing dopants and optionally containing nickel.
INVENTOR(S): Ansmann, Andreas; Benisch, Christoph
PATENT ASSIGNEE(S): BASF AG, Germany
SOURCE: Ger. Offen., 14 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10152135	A1	20030430	DE 2001-10152135	20011023
US 2003120115	A1	20030626	US 2002-271977	20021017
US 6790996	B2	20040914		
EP 1306365	A2	20030502	EP 2002-23640	20021021
EP 1306365	A3	20031015		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK				
JP 2003192647	A2	20030709	JP 2002-307884	20021023

PRIORITY APPLN. INFO.: DE 2001-10152135 A 20011023

OTHER SOURCE(S): CASREACT 138:337704; MARPAT 138:337704

L5 ANSWER 5 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB Some of the largest com. produced primary amines are manufactured by catalytic hydrogenation of nitriles using sponge metal catalysts. The larger the market volume for the amine, the more important the technol. used to control selectivity becomes to remain a viable producer. We have found that controlling the selectivity to the primary amine using lithium hydroxide modified sponge cobalt in backmix reactors, batch, semi-batch or continuous, at moderate pressures and temps. provides an excellent means of minimizing byproducts without sacrificing productivity. LiOH modified sponge cobalt was found to recycle in batch processing without loss of selectivity for primary amines. In continuous backmix processing LiOH modified sponge cobalt catalyst retained selectivity through numerous reactor turnovers compared to LiOH modified sponge nickel. NaOH and KOH modified catalysts tended to agglomerate under similar conditions. Procedures using a semi-batch system are provided for selecting optimum catalysts for nitrile hydrogenation, measuring the catalysts activity and its ability to resist poisoning by nitriles. This paper presents a practical approach to selecting the best selectivity control for the com. production of primary amines and demonstrates that chemical additives alone are not enough to allow one to obtain the best possible control over selectivity and in fact, the mode of operation and reaction conditions are also important in the optimization process.

ACCESSION NUMBER: 2001:439661 CAPLUS
DOCUMENT NUMBER: 136:120171
TITLE: Lithium hydroxide modified sponge catalysts for control of primary amine selectivity in nitrile hydrogenations
AUTHOR(S): Johnson, Thomas A.; Freyberger, Douglas P.
CORPORATE SOURCE: Consultant for Process Development Chemistry, Orefield, PA, 18069, USA
SOURCE: Chemical Industries (Dekker) (2001), 82(Catalysis of Organic Reactions), 201-227
CODEN: CHEIDI; ISSN: 0737-8025
PUBLISHER: Marcel Dekker, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English

L5 ANSWER 6 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB RlCN were transfer hydrogenated using R2CH2NH2 [R1, R2= alkyl, X(CH2)y, (CH2)kNMe2, (CH2)mPh, (CH2)nNH(CH2)n+1NH2, (CH2)pNH(CH2)pCN; x = cyano, H2NCH2; k = 2-17; m = 1-17; n, p = 3-11; yr = 3-16] at 20-200° in the presence of Raney Ni and in the absence of H. Thus, hexanenitrile (I) 3.2 g and octylamine (II) 3.1 g were heated at 100° with 3.4 g Raney Ni for 45 min to give a mixture containing I 31, II 48, hexylamine 8.1, and octylnitrile 2.7 area %.

ACCESSION NUMBER: 1994:30458 CAPLUS
DOCUMENT NUMBER: 120:30458
TITLE: Transfer hydrogenation of nitriles using amine donors
INVENTOR(S): Weigert, Frank J.
PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co., USA
SOURCE: U.S., 5 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5237088	A	19930817	US 1992-857344	19920325

PRIORITY APPLN. INFO.: US 1992-857344 19920325

OTHER SOURCE(S): CASREACT 120:30458; MARPAT 120:30458

L5 ANSWER 7 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB Amines are prepared by hydrogenation of nitriles with metal catalysts prepared by mixing polyalc. solns. of salts of hydrogenating metals with metal alkoxides or silica sol (as materials for supports), treatment with H2O for hydrolysis, drying the resulting gels, optional calcining, and reduction Ni(NO3)2 was dissolved in ethylene glycol, treated with Et silicate at 80° for 3 h, and treated with H2O at 80° for 3 h to give gel, which was dried, calcined at 500° for 3 h, and reduced at 500° for 2 h under H to give Ni catalyst supported on silica. Autoclaving succinonitrile with ammonia and the catalyst at 100° and 20 atm H for 12 min gave 95% 4-aminobutyronitrile.

ACCESSION NUMBER: 1993:516785 CAPLUS
DOCUMENT NUMBER: 119:116785
TITLE: Preparation of amines by hydrogenation of nitriles
INVENTOR(S): Nakamura, Katsumi; Okamoto, Yasushi
PATENT ASSIGNEE(S): Nitto Chemical Industry Co Ltd, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05097776	A2	19930420	JP 1991-289429	19911009
JP 3014192	B2	20000228		

PRIORITY APPLN. INFO.: JP 1991-289429 19911009

OTHER SOURCE(S): CASREACT 119:116785

L5 ANSWER 8 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Keeping 100 ml. 22% aqueous Me2NH. 46 ml. 35% formalin, 0.1 ml. 5N NaOH and 41
ml. Me2C(OH)CN 3 hrs. gave after extraction with CHCl3, 55.2% Me2NCH2CN, b.
133-6°. Similarly was prepared 82.7% (CH2)5NCH2CN, b12 83-4°
[(CH2)5N = piperidino]. CH2:CHCN and 22% aqueous Me2NH overnight gave 74.8%
Me2NCH2CH2CN, b. 171-2°. Reduction of the nitriles with LiAlH4 in Et2O
2 hrs. gave: 65% Me2NCH2CH2NH2, b. 103-5°; and 62% (CH2)5
NCH2CH2NH2., b30 78-80°. Me2N(CH2)3NH2, 63%, b. 136-7°;
Et2N(CH2)3NH2, 67.2%, b25 72°; and (CH2)5NCH2CH2CH2NH2, 81%, b8
80° were prepared by hydrogenation over Raney Ni at
80-100° under 100-20 atmospheric in MeOH-NH3. NaHSO3.CH2O treated with
the above amines in H2O, the mixts. kept 1 hr., then treated with aqueous KCN
2 hrs., gave the following R2N(CH2)n-NHCH2CN (R2N and n shown): Me2N, 2, 35.3%, b40 119°; Et2N, 2, 44%, b38 137-40°; (CH2)5N, 2, 32%,
b6 110-19°; Me2N, 3, 40.6%, b5 104-5°; Et2N, 3, 51%, b4
114°; (CH2)5N, 3, 49%, b2 122-4°. These treated with dry N
oxides in Et2O with cooling 2 hrs. (until blue-green color had formed)
gave an oily precipitate which with Et.2O.HCl gave the following
3-dialkyl-aminoalkylsydnone imines (I) (R and n shown), isolated as di-HCl
salts: Me, 2, m. 165-6°; Et, 2, m. 151°; (R2N =)(CH2)5N, 2,
m. 162-3°; Me, 3, m. 170-1°; Et, 3, m. 162-3°
(isolated as picrate); (R2N=)(CH2)5N, 3, m. 156-7°.
ACCESSION NUMBER: 1963:403479 CAPLUS
DOCUMENT NUMBER: 59:3479
ORIGINAL REFERENCE NO.: 59:602f-h,603a
TITLE: Sydnones and sydnone imines. XV. Synthesis of 3-(dialkylaminoalkyl)syndnone imines
AUTHOR(S): Yashunskii, V. G.
CORPORATE SOURCE: S. Ordzhonikidze All-Union Chem.-Pharm. Res. Inst., Moscow
SOURCE: Zhurnal Obshchei Khimii (1963), 33, 192-5
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L5 ANSWER 9 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. CA 55, 223281; Elslager, et al., CA 51, 1182d. Several dialkylaminoalkylaminopyridines and pyrimidines were prepared
N-Cyanomethylation of the corresponding amines gave N-eyanomethylpiperidine, b. 210°, in 94% yield and Et2NCH2CN, b.
170°, in 80% yield. Gradual addition of 25% Me2NH solution to CH2:CHCN gave 86% Me2NCH2CH2CN, b. 171-2°; picrate m. 155°.
Et2-NCH2CH2CN, b. 195-6°, 92%, piperidinepropionitrile, b.
220-2°, 90%, and morpholinopropionitrile, b. 244-6°, 90%
yield, were prepared by the method of Whitemore, et al., (CA 38, 36173).
Nitriles were reduced with Raney Ni in slightly alkaline solns.
(e.g. 0.1 g. NaOEt/0.1 mole nitrile) to amines: β-piperidinoethylamine, b. 184°, 70% yield; Et2NCH2CH2NH2, b.
145°, 65% yield; Me2NCH2CH2CH2-NH2, b. 125-6°, 65% yield
(picrate m. 220°); Et2NCH2CH2-CH2NH2, b. 168°, 78% yield;
β-piperidinopropylamine, b. 202-4°, 85% yield;
γ-morpholinopropylamine, b. 216-18°, 88% yield. AcCH2CO2Et
(13 g.) and NH2CSNH2 (18 g.) were added to 3 g. Na in 50 ml. alc., the mixture kept 1 hr. at 50°, refluxed 2 hrs., the alc. distilled, the residue dissolved in water, and acidified with AcOH to give
4-methyl-2-thiouracil (95% yield), m. above 270° (AcOH). This in
5% Na2CO3 was treated with Me2SO4 to give 4-methyl-2-methylthiouracil, m. 217-19°. 2-Chloro-5-nitropyridine refluxed in alc. with fused NaOAc and the appropriate amine gave the following 5-nitro-2-(γ-dialkylaminopropylamino)-pyridines (dialkylamino group, % yield, m.p., m.p. of picrate given): Me2N, 70, 64°, --; Et2N, 72, 78-80°,
--; piperidino, 70, 80-2°, 195°; morpholino, 75,
102°, 112°. (These compds. were hygroscopic, m.ps. were determined in sealed tubes.) Heating substituted 2-methylthiopyrimidines with
the appropriate amine at 170 gave the following 6,4-R'-(HO)C4N2NH(CH2)nR''-
2 (n, R', R'', % yield, m.p., m.p. of picrate given): 1, Me, Ph, 55, above
250°, 190°; 1, OH, Ph, 45, above 250°, 204°;
2, Me, OH, 95, 190°, 194°; 2, OH, OH, 80, 172°,
174°; 2, Me, piperidino, 75, above 240°, 175°; 2, Me,
Et2N, 80, above 250°, 244°; 3, Me, Me2N, 65, 68°,
178°; 3, Me, Et2N, 70, 70°, 193°; 3, Me, piperidino,
80, 75°, 210°; 3, Me, morpholino, 75, 98°,
218°. Treating the appropriate 2-dialkylaminoalkylaminopyrimidine in C6H6 with Cl2CHCOCl gave the following 6,4-Me-(HO)C4N2[N(COCHCl2)(CH2)nR]-2 (n, R, % yield, m.p. given) (recrystd. from dimethylformamide): 1, Ph, 40, 190°; 2, OH, 55, 172°; 2,
piperidino, 56, 134°; 3, Et2N, 60, 84°; 3, morpholino, 58,
98°; 3, piperidino, 62. 86°. The appropriate alc. treated
with SOCl2 in C6H6 gave the following ethyl chloride hydrochlorides:
2-piperidino, m. 226°; 2-morpholino, m. 180°. Refluxing
2-(2-hydroxy-ethylamino)-4-methyluracil in C6H6 with NaNH2 and the appropriate dialkylaminoethyl chloride HCl gave the following
6,4-R'(HO)C4N2[N(CH2CH2Cl)CH2CH2R'']-2 (R', R', % yield, m.p. given): OH,
OH, 50, 181°; Me, OH, 50, above 270°; Me, Et2N, 55,
198° (hygroscopic); Me, piperidino, 60, 204° (hygroscopic);
Me, morpholino, 60, 217° (hygroscopic).
ACCESSION NUMBER: 1962:423227 CAPLUS
DOCUMENT NUMBER: 57:23227
ORIGINAL REFERENCE NO.: 57:4662a-g
TITLE: Possible antiamebic agents. XVI
AUTHOR(S): Sen, A. B.; Gupta, S. K.
CORPORATE SOURCE: Univ. Lucknow, India

L5 ANSWER 9 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
SOURCE: J. Indian Chem. Soc. (1962), 39, 129-34
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L5 ANSWER 10 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB R2MeNI(CH2)nNHC(:NH)NH2.HI (I) were prepared, where R was an alkyl radical
or R2N a heterocyclic radical and n = 2-5. To 350 cc. com. aqueous NaHSO3 and
100 cc. 40% aqueous CH2O was added gradually 1 mole amine, 1st at 65° and then at 35° (cooling) with stirring and under reflux (with
highly volatile amines) (the com. aqueous solns. or the anhydrous amines could be
used), the mixture treated during 90 min. with 150 cc. 50% aqueous NaCN, and the
upper nitrile layer decanted, dried, and distilled to give the following
R2N(CH2)nCN (II) (n = 1) (R, % yield, b.p./ mm., m.p. of methiodide given): Me, 71, 138°/.apprx.760, 210°; Et, 67 64°/15,
181°; (R2N =) pyrrolidino, 60, 84-5°/17, 216°; (R2N
=) piperidino, 72.5, 95°/15, 197°. CH2:CHCN (III)
(equimolar amount) added gradually to a secondary amine (com. aqueous solution or
anhydrous diluted with C6H6) below 30°, the mixture stirred 2 hrs., and
the nitrile separated by distillation (the nitriles were salted out when present in
aqueous solution, dried, and distilled) gave the following II (n = 2) (R, solvent, %
yield, and b.p./mm. given): Me, H2O, 90, 72°/19 (HCl salt m.
203°); Et, H2O, quant., 89-90°/16 (HCl salt m. 126°);
(R2N =) pyrrolidino, C6H6, quant., 104-5°/20 (methiodide m.
126°); (R2N =) piperidino, C6H6, 96%, 110-11°/16 (methiodide
m. 156-7°). γ-Butyrolactone (1 mole), 50 cc. MeOH, and an
unsealed ampul containing 80 cc. liquid NH3 placed in a 500 cc. steel autoclave, the contents stirred vigorously, heated 16 hrs. at 100°
(bath temperature), cooled, filtered, the filtrate evaporated in vacuo, the residue
treated with 80 cc. C6H6, and the mixture evaporated on a H2O bath gave 97 g.
crude HO(CH2)3CONH2 (IV). Crude IV (51 g.) in 100 cc. CHCl3 treated
gradually with 130 g. SOCl2 (highly exothermic reaction), when the
reaction subsided the solution boiled until evolution of HCl ceased, and
distilled gave 36 g. Cl(CH2)3CN (V), b15 81°. The anhydrous secondary
amines (2 moles) and 1 mole V in Me2CO heated 24-48 hrs. at 100° in
an autoclave, the precipitate filtered off, and the filtrate fractionated (in the
case of pyrrolidine where its HCl salt was soluble in Me2CO, the Me2CO was
removed on a H2O bath, the base was liberated with alkali, decanted, and
distilled) gave the following II (n = 3) (R, % yield, b.p./mm., m.p. of
methiodide given): Me, 78, 91-2°/18, 203°; Et, 70,
97°/18, 193°; (R2N =) pyrrolidino, 78, 115°/18,
143°; (R2N =) piperidino, 80, 126°/18, 124°.
Pyrolysis of MeCH:CHCH(CN)OBz at 450 ± 10° (method of Snyder et al., CA 43, 4217g) gave 77% Me2NCH2CH:CHCH2CN, b32 53°.
Me2NCH2CH2OH (0.33 mole), 50 cc. C6H6, and 30 drops 40% aqueous Triton B
treated gradually with III with stirring below 25°, the mixture
stirred 2 hrs., neutralized with 2 g. NH4Cl, filtered, and the filtrate distilled gave 90% R2NCH2CH2OCH2CH2CN (VI) (R = Me), b18 114-15°;
methiodide m. 125°. Similarly was prepared 92% VI (R = Et). The
preceding nitriles were reduced (A) chemical with Na in EtOH-PhMe (method of
Bloom, et al., CA 39, 24869) and (B) catalytically (1) in MeOH solution at
90-100° with Raney Co and liquid NH3, (2) in MeOH solution at

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90-100° with Raney Ni, and (3) in MeOH soln. at
60° with Raney Ni to give the following R2N(CH2)nNH2
(VII) [R, n, method, initial pressure (kg./cm.), % yield, b.p. given]:
Me,
2, A, -, -, 108°; Et, 2, A, -, 46, 145°; (R2N =)
pyrrolidino, 2, A, -, 43, 184-5°; (R2N =) piperidino, 2, A, -, 45,
134-5°; Me, 3, B-1, 130, 53, 168°; Et, 3, B-1, 110, 85,
167°; (R2N =) pyrrolidino, 3, B-1, 75, 64, 187°; (R2N =)
piperidino, 3, B-1, 90, 69, b18 89-90°; Me, 4, B-1, 70, 50,
157°; Et, 3, B-1, 72, 59, 189°; (R2N =) pyrrolidino, 4, B-1,
70. 64, 205° (b19 95-7°); (R2N =) piperidino, 4, B-1, 80,
73, 224-5°; Me, 5, B-2, 80, 39, b16 79-80°; Et, 5, B-2, 80,
43, b16 10.3°; and the following R2NCH2CH2O(CH2)3NH2: Me, -, B-3,
70, 52, b23 99-100°; Et, -, B-3, 75, 59, b20 112-13°.
(MeSC(:NH)NH2)2.H2SO4 (0.5 mole), 1.1 moles NaI, and 250 cc. abs. EtOH
refluxed 4 hrs., filtered, the filtrate evapd., the residue treated with
100 cc. Me2CO, the mixt. filtered, the soln. evapd., and the product
washed with cold EtOAc gave 88% MeSC(:NH)NH2.HI (VIII), m. 117°.
VII (R = Me, n = 2) (IX) HCl salt (12 g.) and 16.2 g. VIII added to
NaOEt
soln. (from 3.5 g. Na and 75 cc. EtOH), the mixt. refluxed 45 min.,
evapd., the residue dissolved in 40 cc. Me2CO, the filtered soln. dild.
with an equal vol. of BuOH, and treated gradually with 10.5 g. MeI with
cooling gave I (R = Me, n = 2) (X), m. 181° (Me2CO-MeOH). VII (R =
Et, n = 2) (0.1 mole) and 0.1 mole VIII in 60 cc. abs. EtOH refluxed
until
MeSH ceased to evolve, the EtOH evapd. in vacuo on a H2O bath, the
residue
taken up in 50 cc. Me2CO, the filtered soln. cooled, and treated
gradually with 0.1 mole MeI gave I (R = Et, n = 2), m. 159°
(EtOAc-MeOH). The following I were prepd. by the latter method in 60-80%
yields (R, n, and m.p. given): (R2N =) pyrrolidino, 2, 136°; (R2N
=) piperidino, 2, 136°; Me, 3, 153-4°; Et, 3, 151°;
(R2N =) pyrrolidino, 3, 121°; (R2N =) piperidino, 3, 158°;
Me, 4, 171.5°; Et, 4, 115.5°; (R2N =) pyrrolidino, 4,
131°; (R2N =) piperidino, 4, 156-7°; Me, 5, -; Et, 5,
138-9°. R2MeNICH2CH2O- (CH2)3NHC(:NH)NH2.HI (XI) (R = Me), m.
110°, and XI (R = Et) (dipicrate), were also prepd. Proof of
structure of the I. Application of the Sakaguchi reaction to the I gave
a
pos. reaction, which did not occur with a mono-substituted guanidine. To
a concd. soln. of 0.1 mole BrCH2CH2NH2.HBr in MeOH was added 0.3 mole
anhyd. Me3N (previously chilled), the ppt. collected, the filtrate evapd.
in vacuo, the residual basic oil dissolved in MeOH-iso-PrOH, the soln.
neutralized with HBr, and the product dried to give Me3NBrCH2CH2NH2.HBr
(XII). XII dissolved in a suspension of moist Ag2O (from 0.2 mole AgNO3)
in H2O, the mixt. stirred several min., filtered, the filtrate
neutralized
with HI, evapd. in vacuo, and the residue washed with iso-PrOH-Me2CO gave
Me3NICH2CH2NH2.HI (XIII). XIII (0.05 mole) added to NaOEt soln. (from
0.05 mole Na and 75 cc. abs. EtOH), the soln. treated with 0.05 mole
VIII,
refluxed 2 hrs., and evapd. to 1/3 vol. gave X, m. 181°
(iso-PrOH-MeOH), identical with X prepd. above. To 0.5 mole MeNHCSNH2 in
150 cc. Me2CO was added gradually 0.5 mole MeI with stirring to give 93%
MeSC(:NH)NH-Me.HI (XIV), m. 135° (Me2CO). XIV (0.05 mole) and 0.05
mole IX in 30 cc. EtOH refluxed 30 min., cooled, treated with 0.05 mole
HI
(as 66% soln.), and dild. with EtOAc gave 94% Me2N(CH2)nNHC(:NH)NHMe.2HI

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(XV) (n = 2), m. 142-3° (EtOH-iso-PrOH). Similarly was prepd. XV
(n = 3), m. 121-2°.

ACCESSION NUMBER: 1962:31056 CAPLUS
DOCUMENT NUMBER: 56:31056
ORIGINAL REFERENCE NO.: 56:5830d-i,5831a-h
TITLE: Preparation of guanidines having in addition a quaternary ammonium function
AUTHOR(S): Lespagnol, A.; Cheymol, J.; Cuingnet, E.; Debaert, M.;
Adolphe, M.; Adolphe, C.
CORPORATE SOURCE: Univ. Lille, Fr.
SOURCE: Congr. Sci. Pharm. (1960), 1959, 194-308
DOCUMENT TYPE: Journal
LANGUAGE: French

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AB cf. C.A. 48, 9371b. To 320 g. CH2:CHCN was added gradually 1 kg. 35%
Me2NH at 40-5°; after 40 min. the mixture was saturated with NaOH
yielding 82% Me2NCH2CH2CN, b. 169-72°; similarly was prepared
Et2NCH2CH2CN, b20 86-8°, and (CH2)5NCH2CH2CN, b18 114-5°.
To 225g. CH2:CHCN and a few drops of MeONa-MeOH was added 180 g. MeOH at
40-5°; on the following day acidification with AcOH gave 80.5%
MeOCH2CH2CN, b. 162-4°; similarly were prepared: 78% EtOCH2CH2CN, b.
169-72°; 95% PrOCH2CH2CN, b24 85-9°; 85.5% BuOCH2CH2CN, b10
74-5; 95% EtSCH2CH2CN, b13 100°. These saturated with NH3 in ROH with
ice cooling and hydrogenated over Raney Ni at 95-100° at
90-120 atmospheric H gave: 63.5% MeOCH2CH2CH2NH2, b734 117-19°; 50%
EtOCH2CH2CH2NH2, b. 133-5°; 50% PrOCH2CH2NH2, b. 153-6°; 71%
BuOCH2CH2CH2NH2, b21 74-6°. To 40 g. Na dispersed in MePh was
added 36 g. EtSCH2CH2CN in 200 ml. dry EtOH, followed after dissolution
of
Na by 50 ml. EtOH and 200 ml. H2O; after acidification with HCl,
concentration in
vacuo, washing with Et2O, treatment with solid KOH, and extraction with
Et2O
there was obtained 28% EtSCH2CH2CH2NH2, b23 86-7°, nD20 1.4855, d20
0.9370. Saturation of 465 g. Me2NCH2CH2CN in 500 ml. MeOH with NH3 with
cooling followed by hydrogenation over Raney Ni at 110 atmospheric H as
above gave 68.8% Me2NCH2CH2CH2NH2 (I), b. 130-3°; similarly were
obtained: 65% Et2NCH2CH2CH2NH2, b. 168-70°, and 50%
(CH2)5NCH2CH2CH2NH2, b10 82-5°. To 10 g. I in 10 ml. H2O was added
in 5 min. 13 g. MeCH:CHCOCMe:CH2 (mixed with corresponding methoxy
ketones) in 10 ml. MeOH; after 8.5 hrs. at reflux the mixture was
diluted and
acidified with HCl, concentrated in vacuo, extracted with Et2O, treated
with KOH,
and extracted with Et2O yielding 64%
1-(3-dimethylaminopropyl)-2,5-dimethyl-4-
piperidone, b2.5 96-7°, nD20 1.4726, d2020 0.9369 (di-HCl salt, m.
187-8°); a similar reaction in aqueous MeOH 6 hrs. at room temperature
gave
87% above piperidone, while in MeOH an 8.5 hr. heating gave but 28%
yield.
All the piperidones described in this paper irritate the skin.
Similarly,
Et2NCH2CH2CH2NH2 gave in 8 hrs. at room temperature 77%
1-(3-diethylaminopropyl)-
2,5-dimethyl-4-piperidone, b2 118-20°, 1.4678, 0.9146;
(CH2)5NCH2CH2CH2NH2 similarly gave 57.5% 1-(3-piperidylaminopropyl)-2,5-
dimethyl-4-piperidone, b1 126-7°, 1.4885, 0.9717. Similarly,
MeOCH2CH2CH2NH2 in 4 hrs. in aqueous MeOH at room temperature gave 75%
1-(3-methoxypropyl)-2,5-dimethyl-4-piperidone, b3 110-11°, 1.4547,
0.9564 (HCl salt, m. 133-4°). This heated with N2H4.H2O in aqueous
EtOH 5 hrs. at 70-5°, then freed of solvent, and heated with KOH
fused in an Ag dish to 150-60° gave 53% 1-(3-methoxypropyl)-2,5-
dimethylpiperidine, b2.5 60-2°, 1.4520, 0.8874 (HCl salt, m.
131.5-3°). Similarly were prepared: 68.7% 1-(3-ethoxypropyl)-2,5-
dimethyl-4-piperidone, b2.5 116-18°, 1.4554, 0.9468 (HCl salt,
oil); 41.5% 1-(3-ethoxypropyl-2,5-dimethylpiperidine, b2 58-60°,
1.4524, 0.8790 (HCl salt, oil); 67% 1-(3-propoxypropyl)-2,5-dimethyl-4-
piperidone, b1.5 117-19°, 1.4545, 0.9357 (HCl salt, oil); 65%
1-(3-butoxypropyl)-2,5-dimethyl-4-piperidone, b2 127-9°, 1.4494,
0.9171 (HCl salt, oil); 68% 1-(3-ethylmercaptopropyl)-2,5-dimethyl-4-
piperidone, b1.5 117-18°, 1.4915, 0.9896 (picrate, oil). Keeping
10 g. I, 16 g. 5-methyl-2,5-heptadien-4-one, 10 ml. H2O, and 20 ml. MeOH

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hrs. at room temp. and 40 min. at 50-60° gave 72%
1-(3-dimethylaminopropyl)-2,5,6-trimethyl-4-piperidone, b1.5
104-6°, 1.4742, 0.9420. Similarly were prepd.: 71.5%
1-(3-diethylaminopropyl)-2,5,6-trimethyl-4-piperidone, b1.5
112-13°, 1.4736, 0.9309; 71% 1-(3-methoxypropyl)-2,5,6-trimethyl-4-
piperidone, b1.5 97-8°, 1.4700, 0.9770; 70% 1-(3-ethoxypropyl)-
2,5,6-trimethyl-4-piperidone, b2 110-11°, 1.4672, 0.9633; 73%
1-(3-propoxypropyl)-2,5,6-trimethyl-4-piperidone, b1.5 111-2°,
1.4645, 0.9510; 70% 1-(3-butoxypropyl)-2,5,6-trimethyl-4-piperidone, b2
119-20°, 1.4638, 0.9411. Similar reactions with propenyl
1-cyclohexenyl ketone similarly gave: 80% 1-(3-dimethylaminopropyl)-2-
methyl-4-oxodecahydroquinoline, b2.5 138-40°, 1.4958, 0.9884; 81%
1-(3-diethylaminopropyl)-2-methyl-4-oxodecahydroquinoline, b2
146-7°, 1.4925, 0.9719; 64.5% 1-(3-piperidylpropyl)-2-methyl-4-
oxodecahydroquinoline, b2 159-61°, 1.4963, 0.9854; 82.5%
1-(3-methoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2.5, 134-5°,
1.4951, 1.0219 (picrate, m. 133-5°); 81.7% 1-(3-ethoxypropyl)-2-
methyl-4-oxodecahydroquinoline, b2.5 140-2°, 1.4880, 1.0075 (this
reduced as above with N2H4 gave 74% 1-(3-ethoxypropyl)-2-
methyldecahydroquinoline, b3 119°, 1.4795, 0.9380 (HCl salt, m.
120-2°); 83% 1-(3-propoxypropyl)-2-methyl-4-oxodecahydroquinoline,
b2 142-3°, 1.4885, 0.9940; 74.5% 1-(3-butoxypropyl)-2-methyl-4-
oxodecahydroquinoline, b2.5, 151-2°, 1.4856, 0.9856.

ACCESSION NUMBER: 1957:85717 CAPLUS
DOCUMENT NUMBER: 51:85717
ORIGINAL REFERENCE NO.: 51:15520b-i,15521a-c
TITLE: Heterocyclic compounds. LII. Synthesis of 1-γ-alkoxypropyl-4-piperidones and 1-γ-dialkylaminopropyl-4-piperidones
AUTHOR(S): Nazarov, I. N.; Makin, S. M.
CORPORATE SOURCE: M. V. Lomonosov Inst. Fine Chem. Technol., Moscow
SOURCE: Zhurnal Obshchei Khimii (1957), 27, 499-509
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L5 ANSWER 12 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB Addition of 105 g. CH2:CHCN to 193 g. cyclohexanol and 14 g. 40% KOH with cooling below 30°, followed by stirring 6 h. at room temperature and standing overnight, gave, after neutralization with dilute HCl and filtration of KCl, 208 g. C6H11OCH2CH2CN, b20 130-2°, nD20 1.4586, d20 0.9674; this (35 g.) hydrogenated in MeOH saturated with NH3 over Raney Ni at 95-105° and 145 atmospheric H to 34.5 g. C6H11OCH2CH2CH2NH2, b4.5 72-4°, nD20 1.4646, d20 0.9281 (phenylcarbamide, m. 101.5-2.5°). Similarly 142 g. 2-methylcyclohexanol gave with 70 g. CH2:CHCN and 10 g. 40% KOH, 137.5 g. 2-methylcyclohexyl 2-cyanoethyl ether, b18 134-7°, nD20 1.4564, d20 0.9502, which hydrogenated as above to 2-methylcyclohexyl 3-aminopropyl ether, b3.5 73-4.5°, nD20 1.4603, d20 0.9115 (phenylcarbamide, m. 96-8°). Similarly 114 g. 3-methylcyclohexanol and 53 g. CH2:CHCN gave 109.5 g. 3-methylcyclohexyl 2-cyanoethyl ether, b16 133-6°, nD20 1.4529, d20 0.9458, which hydrogenated to 3-methylcyclohexyl 3-aminopropyl ether, b4 76-8°, nD20 1.4599, d20 0.9118. To 45 g. 1,2,5-trimethyl-4-piperidinol and 3 g. 40% KOH was added 17 g. CH2:CHCN (temperature rise to 35° observed) and the mixture stirred at room temperature 4 h., then allowed to stand overnight; there was obtained 42 g. 1,2,5-trimethyl-4-piperidyl 2-cyanoethyl ether (I), b4 117°, nD20 1.4635, d20 0.9661 (picrate, m. 111-13°), which hydrogenated to 1,2,5-trimethyl-4-piperidyl 3-aminopropyl ether, b8 116-18°, nD20 1.4680, d20 0.9315 (picrate, m. 147-9°). I (20 g.), 50 mL. EtOH, and 30 g. concentrated H2SO4 stirred 18 h. at 90° gave after dilution, neutralization, and extraction with Et2O 20.2 g. 1,2,5-trimethyl-4-piperidyl 2-carbethoxyethyl ether, b4 109-10°, nD20 1.4504, d20 0.9807 (HCl salt, m. 93.5-5°); similar run in MeOH at 70° failed to react in 8 h. To 122 g. MeNH2 in 700 mL. MeOH was added over 2 h. 208 g. CH2:CHCN at below 30°; after stirring 10 h. at room temperature the mixture gave 255.5 g. MeNHCH2CH2CN, b25 86°, nD20 1.4320, and 54 g. MeN(CH2CH2CN)2, b6 162-4°, nD20 1.4606. Heating 21.5 g. MeNHCH2CH2CN with 16 g. CH2:CHCN 16 h. at 90° gave 31.2 g. MeN(CH2CH2CN)2, b5 159.5-60°, nD20 1.4612. Hydrogenation of the latter in MeOH saturated with NH3 over Raney Ni at 90-100° and 100 atmospheric H gave MeN(CH2CH2CH2NH2)2, b4 81-3°, nD20 1.4753, d20 0.9023, along with some 1-methyl-1,5-diazacyclooctane obtained in the low b. fraction [HCl salt, m. 189.5-90° (from EtOH)]. To 21 g. MeNHCH2CH2CN was added with cooling 21.5 g. CH2:CHCO2Me and the mixture gave after 5 days at room temperature 39.3 g. MeN(CH2CH2CN)CH2CH2CO2Me, b6 133°, b4.5 114°, nD20 1.4507, d20 1.0338 [picrate, m. 108-9° (from EtOH)]. Hydrogenation of this in MeOH saturated with NH3 over Raney Ni at 90-100° and 100 atmospheric H gave a viscous mass which polymerized to a rubbery mass. To 270 g. Me2NH and 400 mL. MeOH was added with cooling 318 g. CH2:CHCN at below 30°; after 1 h. at 40-55° and standing overnight the mixture yielded 566.5 g. Me2NCH2CH2CN, b18 68°, b10 60-1°, nD20 1.4282, which hydrogenated to Me2NCH2CH2CH2NH2, b758 131-4°, nD20 1.4398 (HCl salt, m. 183-4°). Heating 127 g. 2,5-dimethyl-4-piperidone with 53 g. CH2:CHCN 3 h. at 95-7° and allowing the mixture to stand overnight gave 104 g. starting material and 31.2 g. 1-(2-cyanoethyl)-2,5-dimethyl-4-piperidone, b5.5 143-5°, nD20 1.4841, d20 1.0345 [HCl salt, m. 166.5-7° (from Me2CO); picrate, m. 136-7° (from Me2CO)]. This hydrogenated to 1-(3-aminopropyl)-2,5-dimethyl-4-aminopiperidine, b3.5 108-10°, nD20 1.4917, d20 0.9475 (picrate, m.

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227.5-8.5°), under conditions stated above.
ACCESSION NUMBER: 1955:15753 CAPLUS
DOCUMENT NUMBER: 49:15753
ORIGINAL REFERENCE NO.: 49:3034c-i,3035a
TITLE: Cyanoethylation of cyclic and heterocyclic alcohols and amines. Hydrogenation and alcoholysis of products of cyanoethylation
AUTHOR(S): Nazarov, I. N.; Shvekhgeimer, G. A.
CORPORATE SOURCE: Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow
SOURCE: Zhurnal Obshchei Khimii (1954), 24, 163-9
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L5 ANSWER 13 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB Compds. useful as wetting agents, detergents, emulsifying agents, germicides, and fungicides are prepared as follows. (HOCH2CH2)2NH 250 is added slowly to CH2:CHCN (I) 126, and the solution heated 2.4 h. on a steam bath and vacuum-distilled at 100° leaves (HOCH2CH2)2NCH2CH2CN (II) 356 g. II 350 hydrogenated at 2000 lb./sq. in. and 115° in the presence of Raney Ni 10 g. and NH3 4.4 mol gives 76% (HOCH2CH2)2N(CH2)3NH2 (III), b8.6 175-87°. III 33 and stearic acid 56.8 in PhMe 50 refluxed 9 h. at 155-61° give C17H35CONH(CH2)3N(CH2CH2OH)2 (IV) 88 g. HCl 22.4 cc. added to IV 105 g. in EtOH 225 cc., and the solution heated to 62°, treated with ethylene oxide (V) 16 g., and heated 4 h. at 100° give a compound useful as an assistant for stripping vat dyes from cellulosic textiles and as a rewetting agent. HCl 90.5 cc. added to C7H15CONH(CH2)3NMe2 250 in EtOH 450, and the solution heated 3 h. at 50° with V 53 gives C7H15CONH(CH2)3N(Cl)(Me2)CH2CH2OH 340 g. I 170 and 25% aqueous Me2NH 545 give Me2NCH2CH2CN 218 g., which is hydrogenated to Me2N(CH2)3NH2 (VI), b. 134°. Me(CH2)12COCl 38 added dropwise to VI 15.5 in C6H6 160 g. and the solution stirred 1 h. gives C13H27CONH(CH2)3NMe2 (VII), b1-2 215°. V 40 added to VII 266 in EtOH 450 g. and HCl 90.5 cc. and the solution heated 3 h. at 80° gives C13H27CONH(CH2)3N(Cl)(CH2CH2OH)Me2. C13H27CONHCH2CH2NMe2 7.4 and ClCH2CH2OH (VIII) 2 g. in EtOH heated 3 h. at 130-40° give C13H27CONHCH2CH2N(Cl)(CH2CH2OH)Me2. Wood rosin acid 221 and VI 100 g. heated at 200-15° give N-(3-dimethylaminopropyl)abietamide (IX). V 12 passed into IX 93 in alc. 93 g. and HCl 20 cc. and the mixture let stand overnight at 46° gives (3-abietoylaminopropyl)dimethyl(2 - hydroxyethyl)ammonium chloride. C17H35CONH(CH2)3NMe2 (X) 206 in EtOH 300 g. adjusted to pH 3.9 with HCl, heated to 40-50°, and treated with V gives C17H35CONH(CH2)3N(Cl)(CH2CH2OH)Me2. C11H23CONH(CH2)3NMe2 468 and VIII 133 heated 2 h. at 125° give C11H23CONH(CH2)3N(Cl)(CH2CH2OH)Me2. X 117 and ClCH2CH2(OH)CH2OH 35 g. stirred 1.5 h. at 125° give C17H35CONH(CH2)3N(Cl)[CH2CH(OH)CH2OH]Me2.
ACCESSION NUMBER: 1952:67119 CAPLUS
DOCUMENT NUMBER: 46:67119
ORIGINAL REFERENCE NO.: 46:11227i,11228a-e
TITLE: Aliphatic amido propyl quaternary ammonium salts
INVENTOR(S): Cook, Elmer W.; Moss, Philip H.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2589674		19520318	US	

L5 ANSWER 14 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB Quaternary ammonium compds. of the type RCONH(CH2)3N(X)R1R2R3, in which R1 is an alkyl group of at least 7 C atoms, R2 an alkyl group of lower mol. weight, R3 an alkyl, aralkyl, or aliphatic olefin, and X an anion, are prepared by reaction of an appropriate tertiary amine with an alkyl halide, dialkyl sulfate, etc. The new compds. are soluble in H2O, practically odorless, relatively nontoxic to man, and are useful as antiseptics, wetting agents, and emulsifiers. An aqueous solution of Me2NH (25%) 545 was treated with CH2:CHCN 170 parts at a temperature below 20°, left standing for 1 hr., mixed with 350 cc. aqueous NaOH (10%), the aqueous layer extracted with Et2O, and the Et2O removed to yield 218 parts of 2-Me2NC2H4CN (I), b22 73-4°. Hydrogenation of I at 100° and 90 atmospheric pressure in the presence of anhydrous NH3 with Raney Ni as catalyst gave N,N-dimethylpropylenediamine (II), b. 134°. A solution of II 15.5 in C6H6 160 was treated with C13H27COCl 38 parts, stirred for 1 hr., washed with aqueous NaOH (10%) and H2O, and distilled in vacuo to give (3-myristoylaminopropyl)dimethylamine (III), b1-2 208-15°. A solution of III 6.2 and PhCH2Cl 3.4 in C6H6 30 parts was refluxed for 4 hrs. and yielded after removal of the solvent (3-myristoylaminopropyl)dimethylbenzyl ammonium chloride, m. 54°; this compound forms a clear 25% solution in H2O and is a germicide effective against Staphylococcus aureus in a dilution of 1:25,000 at 37° in a 5-min. test and has a PhOH coefficient of 277-333; it is also a wetting agent for cotton fabrics. Using the same procedure, (3-lauroylaminopropyl)dimethylbenzylammonium chloride is obtained, which also is a good germicide.
ACCESSION NUMBER: 1949:23628 CAPLUS
DOCUMENT NUMBER: 43:23628
ORIGINAL REFERENCE NO.: 43:4430h-i,4431a-b
TITLE: Quaternary ammonium compounds
INVENTOR(S): Cook, Elmer W.; Moss, Philip H.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2459062		19490111	US	

L5 ANSWER 15 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB (Stearoylaminopropyl)dimethylbenzylammonium chloride was synthesized in 5 steps: (1) 25% aqueous Me2NH 545 g. was added to CH2:CHCN 170 g. below 20°, poured after 1 hr. into 350 cc. 10% aqueous NaOH, and the oily layer plus the ether extract dried and distilled to yield 218 g. of β-(dimethylamino)propionitrile, b22 73-4°. (2) Me2N(CH2)2CN 207 hydrogenated over Raney Ni at 100° and 90 atmospheric in the presence of NH3 72.4 yielded 3-(dimethylamino)propylamine, b760 134°, 204.5 g. (3) C17H35COCl 49 was added dropwise to Me2N(CH2)3NH2 15.5 in C6H6 160 g. and the solution was washed after 1 hr. with 10% aqueous NaOH and H2O and distilled, giving a solid N,N-dimethyl-3-(stearoylamino)propylamine, b1-2 208-15°. (4) C17H35CONH(CH2)3NMe2 0.4 mol. was treated with C2H4O in the presence of 0.93 g. NaOH in tert-BuOH at 65°, and the NaOH neutralized with 1.9 cc. 38% HCl. (5) The product of step (4) was quaternized by reaction with 51 g. PhCH2Cl at 75° 2 hrs.; after filtering and evaporating off the solvent the quaternary amine salt was a crystalline solid at room temperature, soluble in aqueous Na2CO3 or H2O. Similar products are made using capryl, lauroyl or palmitoyl chloride in step (3) or 1-C10H7CH2Cl in step (5).

ACCESSION NUMBER: 1949:13222 CAPLUS
DOCUMENT NUMBER: 43:13222
ORIGINAL REFERENCE NO.: 43:2630i,2631a-c
TITLE: Aliphatic amide-substituted propyl quaternary ammonium compounds
INVENTOR(S): Moss, Philip H.; Cook, Elmer W.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2459088		19490111	US	

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     (FILE 'HOME' ENTERED AT 14:59:21 ON 24 JAN 2005)

     FILE 'CAPLUS' ENTERED AT 14:59:28 ON 24 JAN 2005
L1           302 S 1738-25-6/RN
L2          2673 S 109-55-7/RN
L3            44 S L1 AND L2
L4        787174 S NI OR NICKEL
L5            15 S L3 AND L4

=>  s sponge or Raney
          23424 SPONGE
           5395 SPONGES
          25390 SPONGE
                  (SPONGE OR SPONGES)
          27788 RANEY
              1 RANEYS
          27788 RANEY
                  (RANEY OR RANEYS)
L6        53134 SPONGE OR RANEY

=> s l6 and l4
L7        27598 L6 AND L4

=> s l2 and l1
L8            44 L2 AND L1

=> s l7 and l1
L9            16 L7 AND L1

=> s l7 and l2
L10           28 L7 AND L2

=> d l9 1-16 abs ibib
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L9 ANSWER 1 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN

AB A low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising **sponge nickel** and aqueous alkali is described.

ACCESSION NUMBER: 2004:609968 CAPLUS
DOCUMENT NUMBER: 141:140075
TITLE: Low-pressure hydrogenation process for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile in the presence of a catalyst system comprising **sponge nickel** and aqueous alkali
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 8 pp., Cont.-in-part of U.S. Ser. No. 327,765.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004147784	A1	20040729	US 2003-731733	20031209
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A2 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 141:140075

L9 ANSWER 2 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN

AB A low-pressure hydrogenation process for the production of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile (I) comprises: feeding hydrogen and 3-(dimethylamino)propionitrile into a low-pressure reactor containing a **sponge nickel** catalyst, at least one Group IA alkali metal hydroxide (e.g., potassium hydroxide), and water to form a reaction medium; heating the reaction medium to 70-100°; pressurizing the reactor to 45-500 psig; and hydrogenating the nitrile to form I.

ACCESSION NUMBER: 2004:589527 CAPLUS
DOCUMENT NUMBER: 141:123405
TITLE: Low-pressure catalytic hydrogenation process for the manufacture of 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: PCT Int. Appl., 20 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004060853	A1	20040722	WO 2003-US39447	20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 6660887	B1	20031209	US 2002-327765 20021223
US 2004147784	A1	20040729	US 2003-731733 20031209

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209

OTHER SOURCE(S): CASREACT 141:123405

L9 ANSWER 3 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN

AB A process for the production of 3-(dimethylamino)propylamine (I) in high (>99%) purity from 3-(dimethylamino)propionitrile utilizing a low-pressure hydrogenation process is described which comprises contacting the nitrile with hydrogen at low pressure in the presence of a **sponge nickel** catalyst and ≥1 Goup IA metal hydroxide at 70-100°/45-150 psig. The improvement in the process resides in a combination of carrying out the hydrogenation process at low pressures and temps. in the presence of a catalytic amount of caustic base in order to give a I selectivity of >99.60%.

ACCESSION NUMBER: 2003:961180 CAPLUS
DOCUMENT NUMBER: 140:17730
TITLE: Low-pressure hydrogenation process and catalyst system for the manufacture of high-purity 3-(dimethylamino)propylamine from 3-(dimethylamino)propionitrile
INVENTOR(S): Ward, Gregory J.; Blanchard, Bryan C.
PATENT ASSIGNEE(S): Solutia Inc., USA
SOURCE: U.S., 7 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6660887	B1	20031209	US 2002-327765	20021223
WO 2004060039	A2	20040722	WO 2003-US29721	20030919
WO 2004060039	A3	20040826		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 2004147784	A1	20040729	US 2003-731733 20031209
WO 2004060853	A1	20040722	WO 2003-US39447 20031212

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2002-327765 A 20021223
US 2003-731733 A 20031209

L9 ANSWER 3 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

OTHER SOURCE(S): CASREACT 140:17730
REFERENCE COUNT: 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 4 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN

AB In a process for the continuous hydrogenation of nitrites to primary amines in the liquid phase over a suspended, activated **Raney** catalyst based on an alloy of aluminum and at least one transition metal selected from iron, cobalt and **nickel**, and, if desired, one or more further transition metals selected from titanium, zirconium, chromium and manganese, the hydrogenation is carried out in the absence of ammonia and basic alkali metal compds. or alkaline earth metal compds.

ACCESSION NUMBER: 2002:369029 CAPLUS
DOCUMENT NUMBER: 136:387718
TITLE: Hydrogenation of nitriles into primary amines over **Raney** catalysts
INVENTOR(S): Ansmann, Andreas; Benisch, Christoph; Funke, Frank; Ohlbach, Frank; Merger, Martin
PATENT ASSIGNEE(S): Germany
SOURCE: U.S. Pat. Appl. Publ., 5 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2002058842	A1	20020516	US 2001-987243	20011114
US 6469211	B2	20021022		
DE 10056840	A1	20020523	DE 2000-10056840	20001116
EP 1209146	A1	20020529	EP 2001-126430	20011108
EP 1209146	B1	20040630		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
AT 270264	E	20040715	AT 2001-126430	20011108
JP 2002201163	A2	20020716	JP 2001-350673	20011115

PRIORITY APPLN. INFO.: DE 2000-10056840 A 20001116

L9 ANSWER 5 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN

AB Nitriles are hydrogenated to primary amines over an activated, alpha-Al2O3-containing, macroporous **Raney** catalyst based on an alloy of aluminum and at least one transition metal selected from the group consisting of iron, cobalt and **nickel**, and, if desired, one or more further transition metals selected from the group consisting of titanium, zirconium, chromium and manganese, which is obtainable by a process comprising: (a) preparing a kneadable composition comprising the alloy, a shaping aid, water and a pore former; (b) shaping the kneadable composition to form a shaped body; (c) calcining the shaped body; (d) activating the calcined shaped body by treatment with an aqueous alkali solution; (e) rinsing the shaped catalyst body with aqueous alkali metal hydroxide solution; and (f) rinsing the shaped catalyst body with water.

ACCESSION NUMBER: 2002:369028 CAPLUS
DOCUMENT NUMBER: 136:387717
TITLE: Hydrogenation of nitriles into primary amines over **Raney** catalysts
INVENTOR(S): Ansmann, Andreas; Benisch, Christoph; Funke, Frank; Ohlbach, Frank; Merger, Martin
PATENT ASSIGNEE(S): BASF Aktiendesellschaft, Germany
SOURCE: U.S. Pat. Appl. Publ., 6 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2002058841	A1	20020516	US 2001-985982	20011107
US 6677486	B2	20040113		
DE 10056839	A1	20020523	DE 2000-10056839	20001116
EP 1207149	A1	20020522	EP 2001-125324	20011026
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
JP 2002205975	A2	20020723	JP 2001-347779	20011113

PRIORITY APPLN. INFO.: DE 2000-10056839 A 20001116

REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 6 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN

AB Some of the largest com. produced primary amines are manufactured by catalytic hydrogenation of nitriles using **sponge** metal catalysts. The larger the market volume for the amine, the more important the technol. used to control selectivity becomes to remain a viable producer. We have found that controlling the selectivity to the primary amine using lithium hydroxide modified **sponge** cobalt in backmix reactors, batch, semi-batch or continuous, at moderate pressures and temps. provides an excellent means of minimizing byproducts without sacrificing productivity. LiOH modified **sponge** cobalt was found to recycle in batch processing without loss of selectivity for primary amines. In continuous backmix processing LiOH modified **sponge** cobalt catalyst retained selectivity through numerous reactor turnovers compared to LiOH modified **sponge nickel**. NaOH and KOH modified catalysts tended to agglomerate under similar conditions. Procedures using a semi-batch system are provided for selecting optimum catalysts for nitrile hydrogenation, measuring the catalysts activity and its ability to resist poisoning by nitriles. This paper presents a practical approach to selecting the best selectivity control for the com. production of primary amines and demonstrates that chemical additives alone are not enough to allow one to obtain the best possible control over selectivity and in fact, the mode of operation and reaction conditions are also important in the optimization process.

ACCESSION NUMBER: 2001:439661 CAPLUS
DOCUMENT NUMBER: 136:120171
TITLE: Lithium hydroxide modified **sponge** catalysts for control of primary amine selectivity in nitrile hydrogenations
AUTHOR(S): Johnson, Thomas A.; Freyberger, Douglas P.
CORPORATE SOURCE: Consultant for Process Development Chemistry, Orefield, PA, 18069, USA
SOURCE: Chemical Industries (Dekker) (2001), 82(Catalysis of Organic Reactions), 201-227
CODEN: CHEIDI; ISSN: 0737-8025
PUBLISHER: Marcel Dekker, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English

L9 ANSWER 7 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN

AB RlCN were transfer hydrogenated using R2CH2NH2 [R1, R2= alkyl, X(CH2)y, (CH2)kNMe2, (CH2)mPh, (CH2)nNH(CH2)n+1NH2, (CH2)pNH(CH2)pCN; x = cyano, H2NCH2; k = 2-17; m = 1-17; n, p = 3-11; yr = 3-16] at 20-200° in the presence of **Raney Ni** and in the absence of H. Thus, hexanenitrile (I) 3.2 g and octylamine (II) 3.1 g were heated at 100° with 3.4 g **Raney Ni** for 45 min to give a mixture containing I 31, II 48, hexylamine 8.1, and octylnitrile 2.7 area %.

ACCESSION NUMBER: 1994:30458 CAPLUS
DOCUMENT NUMBER: 120:30458
TITLE: Transfer hydrogenation of nitriles using amine donors
INVENTOR(S): Weigert, Frank J.
PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co., USA
SOURCE: U.S., 5 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5237088	A	19930817	US 1992-857344	19920325

PRIORITY APPLN. INFO.: US 1992-857344 19920325

OTHER SOURCE(S): CASREACT 120:30458; MARPAT 120:30458

L9 ANSWER 8 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN
AB CH2(CN)2 (I) and NCCH2CONH2 (II) were prepared from Me2NCH:CHCN (III), II via intermediate 5-aminoisoxazole (IV). Thus, Me2NCH2CH2CN was dehydrogenated using a catalyst (e.g., Raney Ni) and a H acceptor (e.g., air) to give III, which was heated with NH2OH.HCl in DMF at 65-70° to give IV. IV was isomerized to II by treatment with MeONa in MeOH. III was stirred with H2NOAc.HCl in CH2ClCH2Cl at room temperature, the precipitate was filtered, and the filtrate heated at reflux to give I.

ACCESSION NUMBER: 1976:30491 CAPLUS
DOCUMENT NUMBER: 84:30491
TITLE: 5-Aminoisoxazole from 3-aminoacrylonitrile
INVENTOR(S): Leimgruber, Willy; Weigele, Manfred
PATENT ASSIGNEE(S): Hoffmann-La Roche, Inc., USA
SOURCE: U.S., 5 pp. Division of U.S. 3,810,935.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3917632	A	19751104	US 1974-435918	19740123
US 3709922	A	19730109	US 1970-42545	19700601
US 3810935	A	19740514	US 1972-262880	19720614
PRIORITY APPLN. INFO.:			US 1970-42545	A3 19700601
			US 1972-262880	A3 19720614

L9 ANSWER 9 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Keeping 100 ml. 22% aqueous Me2NH. 46 ml. 35% formalin, 0.1 ml. 5N NaOH and 41 ml. Me2C(OH)CN 3 hrs. gave after extraction with CHCl3, 55.2% Me2NCH2CN, b. 133-6°. Similarly was prepared 82.7% (CH2)5NCH2CN, b12 83-4° [(CH2)5N = piperidino]. CH2:CHCN and 22% aqueous Me2NH overnight gave 74.8% Me2NCH2CH2CN, b. 171-2°. Reduction of the nitriles with LiAlH4 in Et2O 2 hrs. gave: 65% Me2NCH2CH2NH2, b. 103-5°; and 62% (CH2)5 NCH2CH2NH2., b30 78-80°. Me2N(CH2)3NH2, 63%, b. 136-7°; Et2N(CH2)3NH2, 67.2%, b25 72°; and (CH2)5NCH2CH2CH2NH2, 81%, b8 80° were prepared by hydrogenation over Raney Ni at 80-100° under 100-20 atmospheric in MeOH-NH3. NaHSO3.CH2O treated with the above amines in H2O, the mixts. kept 1 hr., then treated with aqueous KCN 2 hrs., gave the following R2N(CH2)n-NHCH2CN (R2N and n shown): Me2N, 2, 35.3%, b40 119°; Et2N, 2, 44%, b38 137-40°; (CH2)5N, 2, 32%, b6 118-19°; Me2N, 3, 40.6%, b5 104-5°; Et2N, 3, 51%, b4 114°; (CH2)5N, 3, 49%, b2 122-4°. These treated with dry N oxides in Et2O with cooling 2 hrs. (until blue-green color had formed) gave an oily precipitate which with Et.2O.HCl gave the following 3-dialkyl-aminoalkylsydnone imines (I) (R and n shown), isolated as di-HCl salts: Me, 2, m. 165-6°; Et, 2, m. 151°; (R2N =)(CH2)5N, 2, m. 162-3°; Me, 3, m. 170-1°; Et, 3, m. 162-3° (isolated as picrate); (R2N=)(CH2)5N, 3, m. 156-7°.

ACCESSION NUMBER: 1963:403479 CAPLUS
DOCUMENT NUMBER: 59:3479
ORIGINAL REFERENCE NO.: 59:602f-h,603a
TITLE: Sydnones and sydnone imines. XV. Synthesis of 3-(dialkylaminoalkyl)sydnone imines
AUTHOR(S): Yashunskii, V. G.
CORPORATE SOURCE: S. Ordzhonikidze All-Union Chem.-Pharm. Res. Inst., Moscow
SOURCE: Zhurnal Obshchei Khimii (1963), 33, 192-5
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L9 ANSWER 10 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. CA 55, 22328i; Elslager, et al., CA 51, 1182d. Several dialkylaminoalkylaminopyridines and pyrimidines were prepared N-Cyanomethylation of the corresponding amines gave N-eyanomethylpiperidine, b. 210°, in 94% yield and Et2NCH2CN, b. 170°, in 80% yield. Gradual addition of 25% Me2NH solution to CH2:CHCN gave 86% Me2NCH2CH2CN, b. 171-2°; picrate m. 155°. Et2-NCH2CH2CN, b. 195-6°, 92%, piperidinepropionitrile, b. 220-2°, 90%, and morpholinopropionitrile, b. 244-6°, 90% yield, were prepared by the method of Whitemore, et al., (CA 38, 36173). Nitriles were reduced with Raney Ni in slightly alkaline solns. (e.g. 0.1 g. NaOEt/0.1 mole nitrile) to amines: β-piperidinoethylamine, b. 184°, 70% yield; Et2NCH2CH2NH2, b. 145°, 65% yield; Me2NCH2CH2CH2-NH2, b. 125-6°, 65% yield (picrate m. 220°); Et2NCH2CH2-CH2NH2, b. 168°, 78% yield; β-piperidinopropylamine, b. 202-4°, 85% yield; γ-morpholinopropylamine, b. 216-18°, 88% yield. AcCH2CO2Et (13 g.) and NH2CSNH2 (18 g.) were added to 3 g. Na in 50 ml. alc., the mixture kept 1 hr. at 50°, refluxed 2 hrs., the alc. distilled, the residue dissolved in water, and acidified with AcOH to give 4-methyl-2-thiouracil (95% yield), m. above 270° (AcOH). This in 5% Na2CO3 was treated with Me2SO4 to give 4-methyl-2-methylthiouracil, m. 217-19°. 2-Chloro-5-nitropyridine refluxed in alc. with fused NaOAc and the appropriate amine gave the following 5-nitro-2-(γ-dialkylaminopropylamino)-pyridines (dialkylamino group, % yield, m.p., m.p. of picrate given): Me2N, 70, 64°, --; Et2N, 72, 78-80°, --; piperidino, 70, 80-2°, 195°; morpholino, 75, 102°, 112°. (These compds. were hygroscopic, m.ps. were determined in sealed tubes.) Heating substituted 2-methylthiopyrimidines with the appropriate amine at 170 gave the following 6,4-R'-(HO)C4N2NH(CH2)nR''- 2 (n, R', R'', % yield, m.p., m.p. of picrate given): 1, Me, Ph, 55, above 250°, 190°; 1, OH, Ph, 45, above 250°, 204°; 2, Me, OH, 95, 190°, 194°; 2, OH, OH, 80, 172°, 174°; 2, Me, piperidino, 75, above 240°, 175°; 2, Me, Et2N, 80, above 250°, 244°; 3, Me, Me2N, 65, 68°, 178°; 3, Me, Et2N, 70, 70°, 193°; 3, Me, piperidino, 80, 75°, 210°; 3, Me, morpholino, 75, 98°, 210°. Treating the appropriate 2-dialkylaminoalkylaminopyrimidine in C6H6 with Cl2CHCOCl gave the following 6,4-Me-(HO)C4N2[N(COCHCl2)(CH2)nR]-2 (n, R, % yield, m.p. given) (recrystd. from dimethylformamide): 1, Ph, 40, 190°; 2, OH, 55, 172°; 2, piperidino, 56, 134°; 3, Et2N, 60, 84°; 3, morpholino, 58, 98°; 3, piperidino, 62. 86°. The appropriate alc. treated with SOCl2 in C6H6 gave the following ethyl chloride hydrochlorides: 2-piperidino, m. 226°; 2-morpholino, m. 180°. Refluxing 2-(2-hydroxy-ethylamino)-4-methyluracil in C6H6 with NaNH2 and the appropriate dialkylaminoethyl chloride HCl gave the following 6,4-R'(HO)C4N2[N(CH2CH2Cl)CH2CH2R'']-2 (R', R', % yield, m.p. given): OH, OH, 50, 181°; Me, OH, 50, above 270°; Me, Et2N, 55, 198° (hygroscopic); Me, piperidino, 60, 204° (hygroscopic); Me, morpholino, 60, 217° (hygroscopic).

ACCESSION NUMBER: 1962:423227 CAPLUS
DOCUMENT NUMBER: 57:23227
ORIGINAL REFERENCE NO.: 57:4662a-g
TITLE: Possible antiamebic agents. XVI
AUTHOR(S): Sen, A. B.; Gupta, S. K.
CORPORATE SOURCE: Univ. Lucknow, India

L9 ANSWER 10 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
SOURCE: J. Indian Chem. Soc. (1962), 39, 129-34
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L9 ANSWER 11 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN
AB R2MeNI(CH2)nNHC(:NH)NH2.HI (I) were prepared, where R was an alkyl radical
or R2N a heterocyclic radical and n = 2-5. To 350 cc. com. aqueous NaHSO3 and
100 cc. 40% aqueous CH2O was added gradually 1 mole amine, 1st at 65° and then at 35° (cooling) with stirring and under reflux (with highly volatile amines) (the com. aqueous solns. or the anhydrous amines could be
used), the mixture treated during 90 min. with 150 cc. 50% aqueous NaCN, and the
upper nitrile layer decanted, dried, and distilled to give the following R2N(CH2)nCN (II) (n = 1) (R, % yield, b.p./ mm., m.p. of methiodide given): Me, 71, 138°/.apprx.760, 210°; Et, 67 64°/15, 181°; (R2N =) pyrrolidino, 60, 84-5°/17, 216°; (R2N =) piperidino, 72.5, 95°/15, 197°. CH2:CHCN (III)
(equimolar amount) added gradually to a secondary amine (com. aqueous solution or
anhydrous diluted with C6H6) below 30°, the mixture stirred 2 hrs., and the nitrile separated by distillation (the nitriles were salted out when present in
aqueous solution, dried, and distilled) gave the following II (n = 2) (R, solvent, %
yield, and b.p./mm. given): Me, H2O, 90, 72°/19 (HCl salt m. 203°); Et, H2O, quant., 89-90°/16 (HCl salt m. 126°); (R2N =) pyrrolidino, C6H6, quant., 104-5°/20 (methiodide m. 126°); (R2N =) piperidino, C6H6, 96%, 110-11°/16 (methiodide m. 156-7°). γ-Butyrolactone (1 mole), 50 cc. MeOH, and an
unsealed ampul containing 80 cc. liquid NH3 placed in a 500 cc. steel autoclave, the contents stirred vigorously, heated 16 hrs. at 100° (bath temperature), cooled, filtered, the filtrate evaporated in vacuo, the residue
treated with 80 cc. C6H6, and the mixture evaporated on a H2O bath gave 97 g.
crude HO(CH2)3CONH2 (IV). Crude IV (51 g.) in 100 cc. CHCl3 treated gradually with 130 g. SOCl2 (highly exothermic reaction), when the reaction subsided the solution boiled until evolution of HCl ceased, and distilled gave 36 g. Cl(CH2)3CN (V), b15 81°. The anhydrous secondary amines (2 moles) and 1 mole V in Me2CO heated 24-48 hrs. at 100° in an autoclave, the precipitate filtered off, and the filtrate fractionated (in the
case of pyrrolidine where its HCl salt was soluble in Me2CO, the Me2CO was
removed on a H2O bath, the base was liberated with alkali, decanted, and distilled) gave the following II (n = 3) (R, % yield, b.p./mm., m.p. of methiodide given): Me, 78, 91-2°/18, 203°; Et, 70, 97°/18, 193°; (R2N =) pyrrolidino, 78, 115°/18, 143°; (R2N =) piperidino, 80, 126°/18, 124°.
Pyrolysis of MeCH:CHCH(CN)OBz at 450 ± 10° (method of Snyder et al., CA 43, 4217g) gave 77% Me2NCH2CH:CHCH2CN, b32 53°.
Me2NCH2CH2OH (0.33 mole), 50 cc. C6H6, and 30 drops 40% aqueous Triton B treated gradually with III with stirring below 25°, the mixture stirred 2 hrs., neutralized with 2 g. NH4Cl, filtered, and the filtrate distilled gave 90% R2NCH2CH2OCH2CH2CN (VI) (R = Me), b18 114-15°; methiodide m. 125°. Similarly was prepared 92% VI (R = Et). The
preceding nitriles were reduced (A) chemical with Na in EtOH-PhMe (method of
Bloom, et al., CA 39, 24869) and (B) catalytically (1) in MeOH solution at
90-100° with **Raney** Co and liquid NH3, (2) in MeOH solution

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at 90-100° with **Raney Ni**, and (3) in MeOH soln.
at 60° with **Raney Ni** to give the following
R2N(CH2)nNH2 (VII) (R, n, method, initial pressure (kg./cm.), % yield, b.p. given]: Me, 2, A, -, -, 108°; Et, 2, A, -, 46, 145°; (R2N =) pyrrolidino, 2, A, -, 43, 184-5°; (R2N =) piperidino, 2, A, -, 45, 134-5°; Me, 3, B-1, 130, 53, 168°; Et, 3, B-1, 110, 85, 167°; (R2N =) pyrrolidino, 3, B-1, 75, 64, 187°; (R2N =) piperidino, 3, B-1, 90, 69, b18 89-90°; Me, 4, B-1, 70, 50, 157°; Et, 3, B-1, 72, 59, 189°; (R2N =) pyrrolidino, 4, B-1, 70. 64, 205° (b19 95-7°); (R2N =) piperidino, 4, B-1, 80, 73, 224-5°; Me, 5, B-2, 80, 39, b16 79-80°; Et, 5, B-2, 80, 43, b16 10.3°; and the following R2NCH2CH2O(CH2)3NH2: Me, -, B-3, 70, 52, b23 99-100°; Et, -, B-3, 75, 59, b20 112-13°.
[MeSC(:NH)NH2]2.H2SO4 (0.5 mole), 1.1 moles NaI, and 250 cc. abs. EtOH refluxed 4 hrs., filtered, the filtrate evapd., the residue treated with 100 cc. Me2CO, the mixt. filtered, the soln. evapd., and the product washed with cold EtOAc gave 88% MeSC(:NH)NH2.HI (VIII), m. 117°.
VII (R = Me, n = 2) (IX) HCl salt (12 g.) and 16.2 g. VIII added to NaOEt
soln. (from 3.5 g. Na and 75 cc. EtOH), the mixt. refluxed 45 min., evapd., the residue dissolved in 40 cc. Me2CO, the filtered soln. dild. with an equal vol. of BuOH, and treated gradually with 10.5 g. MeI with cooling gave I (R = Me, n = 2) (X), m. 181° (Me2CO-MeOH). VII (R = Et, n = 2) (0.1 mole) and 0.1 mole VIII in 60 cc. abs. EtOH refluxed until
MeSH ceased to evolve, the EtOH evapd. in vacuo on a H2O bath, the residue
taken up in 50 cc. Me2CO, the filtered soln. cooled, and treated gradually with 0.1 mole MeI gave I (R = Et, n = 2), m. 159° (EtOAc-MeOH). The following I were prepd. by the latter method in 60-80% yields (R, n, and m.p. given): (R2N =) pyrrolidino, 2, 136°; (R2N =) piperidino, 2, 136°; Me, 3, 153-4°; Et, 3, 151°; (R2N =) pyrrolidino, 3, 121°; (R2N =) piperidino, 3, 158°; Me, 4, 171.5°; Et, 4, 115.5°; (R2N =) pyrrolidino, 4, 131°; (R2N =) piperidino, 4, 156-7°; Me, 5, -; Et, 5, 138-9°. R2MeNICH2CH2O- (CH2)3NHC(:NH)NH2.HI (XI) (R = Me), m. 110°, and XI (R = Et) (dipicrate), were also prepd. Proof of structure of the I. Application of the Sakaguchi reaction to the I gave a
pos. reaction, which did not occur with a mono-substituted guanidine. To a concd. soln. of 0.1 mole BrCH2CH2NH2.HBr in MeOH was added 0.3 mole anhyd. Me3N (previously chilled), the ppt. collected, the filtrate evapd. in vacuo, the residual basic oil dissolved in MeOH-iso-PrOH, the soln. neutralized with HBr, and the product dried to give Me3NBrCH2CH2NH2.HBr (XII). XII dissolved in a suspension of moist Ag2O (from 0.2 mole AgNO3) in H2O, the mixt. stirred several min., filtered, the filtrate neutralized
with HI, evapd. in vacuo, and the residue washed with iso-PrOH-Me2CO gave Me3NICH2CH2NH2.HI (XIII). XIII (0.05 mole) added to NaOEt soln. (from 0.05 mole Na and 75 cc. abs. EtOH), the soln. treated with 0.05 mole VIII,
refluxed 2 hrs., and evapd. to 1/3 vol. gave X, m. 181° (iso-PrOH-MeOH), identical with X prepd. above. To 0.5 mole MeNHCSNH2 in 150 cc. Me2CO was added gradually 0.5 mole MeI with stirring to give 93% MeSC(:NH)NH-Me.HI (XIV), m. 135° (Me2CO). XIV (0.05 mole) and 0.05 mole IX in 30 cc. EtOH refluxed 30 min., cooled, treated with 0.05 mole HI
(as 66% soln.), and dild. with EtOAc gave 94% Me2N(CH2)nNHC(:NH)NHMe.2HI (XV) (n = 2), m. 142-3° (EtOH-iso-PrOH). Similarly was prepd. XV

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(n = 3), m. 121-2°.

ACCESSION NUMBER:	1962:31056 CAPLUS
DOCUMENT NUMBER:	56:31056
ORIGINAL REFERENCE NO.:	56:5830d-i,5831a-h
TITLE:	Preparation of guanidines having in addition a quaternary ammonium function
AUTHOR(S):	Lespagnol, A.; Cheymol, J.; Cuingnet, E.; Debaert, M.; Adolphe, M.; Adolphe, C.
CORPORATE SOURCE:	Univ. Lille, Fr.
SOURCE:	Congr. Sci. Pharm. (1960), 1959, 194-308
DOCUMENT TYPE:	Journal
LANGUAGE:	French

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AB cf. C.A. 48, 9371b. To 320 g. CH2:CHCN was added gradually 1 kg. 35% Me2NH at 40-5°; after 40 min. the mixture was saturated with NaOH yielding 82% Me2NCH2CH2CN, b. 169-72°; similarly was prepared Et2NCH2CH2CN, b20 86-8°, and (CH2)5NCH2CH2CN, b18 114-5°.
To 225g. CH2:CHCN and a few drops of MeONa-MeOH was added 180 g. MeOH at 40-5°; on the following day acidification with AcOH gave 80.5% MeOCH2CH2CN, b. 162-4°; similarly were prepared: 78% EtOCH2CH2CN, b. 169-72°; 95% PrOCH2CH2CN, b24 85-9°; 85.5% BuOCH2CH2CN, b10 74-5% 95% EtSCH2CH2CN, b13 100°. These saturated with NH3 in ROH with ice cooling and hydrogenated over **Raney Ni** at 95-100° at 90-120 atmospheric H gave: 63.5% MeOCH2CH2CH2NH2, b73 117-19°; 50% EtOCH2CH2CH2NH2, b. 133-5°; 50% PrOCH2CH2NH2, b. 153-6°; 71% BuOCH2CH2CH2NH2, b21 74-6°. To 40 g. Na dispersed in MePh was added 36 g. EtSCH2CH2CN in 200 ml. dry EtOH, followed after dissolution of Na by 50 ml. EtOH and 200 ml. H2O; after acidification with HCl, concentration in vacuo, washing with Et2O, treatment with
solid KOH, and extraction with Et2O there was obtained 28% EtSCH2CH2CH2NH2, b23
86-7°, nD20 1.4855, d20 0.9370. Saturation of 465 g. Me2NCH2CH2CN in 500 ml. MeOH with NH3 with cooling followed by hydrogenation over **Raney Ni** at 110 atmospheric H as above gave 68.8% Me2NCH2CH2CH2NH2 (I), b. 130-3°; similarly were obtained: 65% Et2NCH2CH2CH2NH2, b. 168-70°, and 50% (CH2)5NCH2CH2CH2NH2, b10 82-5°. To 10 g. I in 10 ml. H2O was added in 5 min. 13 g. MeCH:CHCOCMe:CH2 (mixed with corresponding methoxy ketones) in 10 ml. MeOH; after 8.5 hrs. at reflux the mixture was diluted and acidified with HCl,
concentrated in vacuo, extracted with Et2O, treated with KOH, and extracted with Et2O
yielding 64% 1-(3-dimethylaminopropyl)-2,5-dimethyl-4-piperidone, b2.5 96-7°, nD20 1.4726, d2020 0.9369 (di-HCl salt, m. 187-8°); a similar reaction in aqueous MeOH 6 hrs. at room temperature gave 87% above
piperidone, while in MeOH an 8.5 hr. heating gave but 28% yield. All the piperidones described in this paper irritate the skin. Similarly, Et2NCH2CH2CH2NH2 gave in 8 hrs. at room temperature 77% 1-(3-diethylaminopropyl)-
2,5-dimethyl-4-piperidone, b2 118-20°, 1.4678, 0.9146; (CH2)5NCH2CH2CH2NH2 similarly gave 57.5% 1-(3-piperidylaminopropyl)-2,5-dimethyl-4-piperidone, b1 126-7°, 1.4885, 0.9717. Similarly, MeOCH2CH2CH2NH2 in 4 hrs. in aqueous MeOH at room temperature gave 75% 1-(3-methoxypropyl)-2,5-dimethyl-4-piperidone, b3 110-11°, 1.4547, 0.9564 (HCl salt, m. 133-4°). This heated with N2H4.H2O in aqueous EtOH 5 hrs. at 70-5°, then freed of solvent, and heated with KOH fused in an Ag dish to 150-60° gave 53% 1-(3-methoxypropyl)-2,5-dimethylpiperidine, b2.5 60-2°, 1.4520, 0.8874 (HCl salt, m. 131.5-3°). Similarly were prepared: 68.7% 1-(3-ethoxypropyl)-2,5-dimethyl-4-piperidone, b2.5 116-18°, 1.4554, 0.9468 (HCl salt, oil); 41.5% 1-(3-ethoxypropyl-2,5-dimethylpiperidine, b2 58-60°, 1.4524, 0.8790 (HCl salt, oil); 67% 1-(3-propoxypropyl)-2,5-dimethyl-4-piperidone, b1.5 117-19°, 1.4545, 0.9357 (HCl salt, oil); 65% 1-(3-butoxypropyl)-2,5-dimethyl-4-piperidone, b2 127-9°, 1.4494, 0.9171 (HCl salt, oil); 68% 1-(3-ethylmercaptopropyl)-2,5-dimethyl-4-piperidone, b1.5 117-18°, 1.4915, 0.9896 (picrate, oil). Keeping 10 g. I, 16 g. 5-methyl-2,5-heptadien-4-one, 10 ml. H2O, and 20 ml. MeOH 6
hrs. at room temperature and 40 min. at 50-60° gave 72% 1-(3-dimethylaminopropyl)-2,5,6-trimethyl-4-piperidone, b1.5

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104-6°, 1.4742, 0.9420. Similarly were prepd.: 71.5% 1-(3-diethylaminopropyl)-2,5,6-trimethyl-4-piperidone, b1.5 112-13°, 1.4736, 0.9309; 71% 1-(3-methoxypropyl)-2,5,6-trimethyl-4-piperidone, b1.5 97-8°, 1.4700, 0.9770; 70% 1-(3-ethoxypropyl)-2,5,6-trimethyl-4-piperidone, b2 110-11°, 1.4672, 0.9633; 73% 1-(3-propoxypropyl)-2,5,6-trimethyl-4-piperidone, b1.5 111-2°, 1.4645, 0.9510; 70% 1-(3-butoxypropyl)-2,5,6-trimethyl-4-piperidone, b2 119-20°, 1.4638, 0.9411. Similar reactions with propenyl 1-cyclohexenyl ketone similarly gave: 80% 1-(3-dimethylaminopropyl)-2-methyl-4-oxodecahydroquinoline, b2.5 138-40°, 1.4958, 0.9884; 81% 1-(3-diethylaminopropyl)-2-methyl-4-oxodecahydroquinoline, b2 146-7°, 1.4925, 0.9719; 64.5% 1-(3-piperidylpropyl)-2-methyl-4-oxodecahydroquinoline, b2 159-61°, 1.4963, 0.9854; 82.5% 1-(3-methoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2.5, 134-5°, 1.4951, 1.0219 (picrate, m. 133-5°); 81.7% 1-(3-ethoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2.5 140-2°, 1.4880, 1.0075 (this reduced as above with N2H4 gave 74% 1-(3-ethoxypropyl)-2-methyldecahydroquinoline, b3 119°, 1.4795, 0.9380 (HCl salt, m. 120-2°); 83% 1-(3-propoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2 142-3°, 1.4885, 0.9940; 74.5% 1-(3-butoxypropyl)-2-methyl-4-oxodecahydroquinoline, b2.5, 151-2°, 1.4856, 0.9856.

ACCESSION NUMBER: 1957:85717 CAPLUS
DOCUMENT NUMBER: 51:85717
ORIGINAL REFERENCE NO.: 51:15520b-i,15521a-c
TITLE: Heterocyclic compounds. LII. Synthesis of 1-γ-alkoxypropyl-4-piperidones and 1-γ-dialkylaminoropyl-4-piperidones
AUTHOR(S): Nazarov, I. N.; Makin, S. M.
CORPORATE SOURCE: M. V. Lomonosov Inst. Fine Chem. Technol., Moscow
SOURCE: Zhurnal Obshchei Khimii (1957), 27, 499-509
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L9 ANSWER 13 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN
AB Addition of 105 g. CH2:CHCN to 193 g. cyclohexanol and 14 g. 40% KOH with cooling below 30°, followed by stirring 6 h. at room temperature and standing overnight, gave, after neutralization with dilute HCl and filtration of KCl, 208 g. C6H11OCH2CH2CN, b20 130-2°, nD20 1.4586, d20 0.9674; this (35 g.) hydrogenated in MeOH saturated with NH3 over **Raney Ni** at 95-105° and 145 atmospheric H to 34.5 g. C6H11OCH2CH2CH2NH2, b4.5 72-4°, nD20 1.4646, d20 0.9281 (phenylcarbamide, m. 101.5-2.5°). Similarly 142 g. 2-methylcyclohexanol gave with 70 g. CH2:CHCN and 10 g. 40% KOH, 137.5 g. 2-methylcyclohexyl 2-cyanoethyl ether, b18 134-7°, nD20 1.4564, d20 0.9502, which hydrogenated as above to 2-methylcyclohexyl 3-aminopropyl ether, b3.5 73-4.5°, nD20 1.4603, d20 0.9115 (phenylcarbamide, m. 96-8°). Similarly 114 g. 3-methylcyclohexanol and 53 g. CH2:CHCN gave 109.5 g. 3-methylcyclohexyl 2-cyanoethyl ether, b16 133-6°, nD20 1.4529, d20 0.9458, which hydrogenated to 3-methylcyclohexyl 3-aminopropyl ether, b4 76-8°, nD20 1.4599, d20 0.9118. To 45 g. 1,2,5-trimethyl-4-piperidinol and 3 g. 40% KOH was added 17 g. CH2:CHCN (temperature rise to 35° observed) and the mixture stirred at room temperature 4 h., then allowed to stand overnight; there was obtained 42 g. 1,2,5-trimethyl-4-piperidyl 2-cyanoethyl ether (I), b4 117°, nD20 1.4635, d20 0.9661 (picrate, m. 111-13°), which hydrogenated to 1,2,5-trimethyl-4-piperidyl 3-aminopropyl ether, b8 116-18°, nD20 1.4680, d20 0.9315 (picrate, m. 147-9°). I (20 g.), 50 mL. EtOH, and 30 g. concentrated H2SO4 stirred 18 h. at 90° gave after dilution, neutralization, and extraction with Et2O 20.2 g. 1,2,5-trimethyl-4-piperidyl 2-carbethoxyethyl ether, b4 109-10°, nD20 1.4504, d20 0.9807 (HCl salt, m. 93.5-5°); similar run in MeOH at 70° failed to react in 8 h. To 122 g. MeNH2 in 700 mL. MeOH was added over 2 h. 208 g. CH2:CHCN at below 30°; after stirring 10 h. at room temperature the mixture gave 255.5 g. MeNHCH2CH2CN, b25 86°, nD20 1.4320, and 54 g. MeN(CH2CH2CN)2, b6 162-4°, nD20 1.4606. Heating 21.5 g. MeNHCH2CH2CN with 16 g. CH2:CHCN 16 h. at 90° gave 31.2 g. MeN(CH2CH2CN)2, b5 159.5-60°, nD20 1.4612. Hydrogenation of the latter in MeOH saturated with NH3 over **Raney Ni** at 90-100° and 100 atmospheric H gave MeN(CH2CH2CH2NH2)2, b4 81-3°, nD20 1.4753, d20 0.9023, along with some 1-methyl-1,5-diazacyclooctane obtained in the low b. fraction [HCl salt, m. 189.5-90° (from EtOH)]. To 21 g. MeNHCH2CH2CN was added with cooling 21.5 g. CH2:CHCO2Me and the mixture gave after 5 days at room temperature 39.3 g. MeN(CH2CH2CN)CH2CH2CO2Me, b6 133°, b4.5 114°, nD20 1.4507, d20 1.0338 [picrate, m. 108-9° (from EtOH)]. Hydrogenation of this in MeOH saturated with NH3 over **Raney Ni** at 90-100° and 100 atmospheric H gave a viscous mass which polymerized to a rubbery mass. To 270 g. Me2NH and 400 mL. MeOH was added with cooling 318 g. CH2:CHCN at below 30°; after 1 h. at 40-55° and standing overnight the mixture yielded 566.5 g. Me2NCH2CH2CN, b18 68°, b10 60-1°, nD20 1.4282, which hydrogenated to Me2NCH2CH2CH2NH2, b758 131-4°, nD20 1.4398 (HCl salt, m. 183-4°). Heating 127 g. 2,5-dimethyl-4-piperidone with 53 g. CH2:CHCN 3 h. at 95-7° and allowing the mixture to stand overnight gave 104 g. starting material and 31.2 g. 1-(2-cyanoethyl)-2,5-dimethyl-4-piperidone, b5.5 143-5°, nD20 1.4841, d20 1.0345 [HCl salt, m. 166.5-7° (from Me2CO); picrate, m. 136-7° (from Me2CO)]. This hydrogenated to 1-(3-aminopropyl)-2,5-dimethyl-4-aminopiperidine, b3.5 108-10°, nD20 1.4917, d20 0.9475 (picrate, m. 227.5-8.5°), under conditions

L9 ANSWER 13 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
stated above.

ACCESSION NUMBER: 1955:15753 CAPLUS
DOCUMENT NUMBER: 49:15753
ORIGINAL REFERENCE NO.: 49:3034c-i,3035a
TITLE: Cyanoethylation of cyclic and heterocyclic alcohols and amines. Hydrogenation and alcoholysis of products of cyanoethylation
AUTHOR(S): Nazarov, I. N.; Shvekhgeimer, G. A.
CORPORATE SOURCE: Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow
SOURCE: Zhurnal Obshchei Khimii (1954), 24, 163-9
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L9 ANSWER 14 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN
AB Compds. useful as wetting agents, detergents, emulsifying agents, germicides, and fungicides are prepared as follows. (HOCH2CH2)2NH 250 is added slowly to CH2:CHCN (I) 126, and the solution heated 2.4 h. on a steam bath and vacuum-distilled at 100° leaves (HOCH2CH2)2NCH2CH2CN (II) 356 g. II 350 hydrogenated at 2000 lb./sq. in. and 115° in the presence of **Raney Ni** 10 g. and NH3 4.4 mol gives 76% (HOCH2CH2)2N(CH2)3NH2 (III), b8.6 175-87°. III 33 and stearic acid 56.8 in PhMe 50 refluxed 9 h. at 155-61° give C17H35CONH(CH2)3N(CH2CH2OH)2 (IV) 88 g. HCl 22.4 cc. added to IV 105 g. in EtOH 225 cc., and the solution heated to 62°, treated with ethylene oxide (V) 16 g., and heated 4 h. at 100° give a compound useful as an assistant for stripping vat dyes from cellulosic textiles and as a rewetting agent. HCl 90.5 cc. added to C7H15CONH(CH2)3NMe2 250 in EtOH 450, and the solution heated 3 h. at 50° with V 53 gives C7H15CONH(CH2)3N(Cl)(Me2)CH2CH2OH 340 g. I 170 and 25% aqueous Me2NH 545 give Me2NCH2CH2CN 218 g., which is hydrogenated to Me2N(CH2)3NH2 (VI), b. 134°. Me(CH2)12COCl 38 added dropwise to VI 15.5 in C6H6 160 g. and the solution stirred 1 h. gives C13H27CONH(CH2)3NMe2 (VII), b1-2 215°. V 40 added to VII 266 in EtOH 450 g. and HCl 90.5 cc. and the solution heated 3 h. at 80° gives C13H27CONH(CH2)3N(Cl)(CH2CH2OH)Me2. C13H27CONHCH2CH2NMe2 7.4 and ClCH2CH2OH (VIII) 2 g. in EtOH heated 3 h. at 130-40° give C13H27CONHCH2CH2N(Cl)(CH2CH2OH)Me2. Wood rosin acid 221 and VI 100 g. heated at 200-15° give N-(3-dimethylaminopropyl)abietamide (IX). V 12 passed into IX 93 in alc. 93 g. and HCl 20 cc. and the mixture let stand overnight at 46° gives (3-abietoylaminopropyl)dimethyl(2 - hydroxyethyl)ammonium chloride. C17H35CONH(CH2)3NMe2 (X) 206 in EtOH 300 g. adjusted to pH 3.9 with HCl, heated to 40-50°, and treated with V gives C17H35CONH(CH2)3N(Cl)(CH2CH2OH)Me2. C11H23CONH(CH2)3NMe2 468 and VIII 133 heated 2 h. at 125° give C11H23CONH(CH2)3N(Cl)(CH2CH2OH)Me2. X 117 and ClCH2CH2(OH)CH2OH 35 g. stirred 1.5 h. at 125° give C17H35CONH(CH2)3N(Cl)[CH2CH(OH)CH2OH]Me2.

ACCESSION NUMBER: 1952:67119 CAPLUS
DOCUMENT NUMBER: 46:67119
ORIGINAL REFERENCE NO.: 46:11227i,11228a-e
TITLE: Aliphatic amido propyl quaternary ammonium salts
INVENTOR(S): Cook, Elmer W.; Moss, Philip H.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2589674		19520318	US	

L9 ANSWER 15 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN
AB Quaternary ammonium compds. of the type RCONH(CH2)3N(X)R1R2R3, in which R1
is an alkyl group of at least 7 C atoms, R2 an alkyl group of lower mol. weight, R3 an alkyl, aralkyl, or aliphatic olefin, and X an anion, are prepared
by reaction of an appropriate tertiary amine with an alkyl halide, dialkyl
sulfate, etc. The new compds. are soluble in H2O, practically odorless, relatively nontoxic to man, and are useful as antiseptics, wetting agents,
and emulsifiers. An aqueous solution of Me2NH (25%) 545 was treated with CH2:CHCN 170 parts at a temperature below 20°, left standing for 1 hr., mixed with 350 cc. aqueous NaOH (10%), the aqueous layer extracted with Et2O, and the
Et2O removed to yield 218 parts of 2-Me2NC2H4CN (I), b22 73-4°. Hydrogenation of I at 100° and 90 atmospheric pressure in the presence of anhydrous NH3 with **Raney Ni** as catalyst gave
N,N-dimethylpropylenediamine (II), b. 134°. A solution of II 15.5 in C6H6 160 was treated with C13H27COCl 38 parts, stirred for 1 hr., washed with aqueous NaOH (10%) and H2O, and distilled in vacuo to give (3-myristoylaminopropyl)dimethylamine (III), b1-2 208-15°. A solution of III 6.2 and PhCH2Cl 3.4 in C6H6 30 parts was refluxed for 4 hrs. and yielded after removal of the solvent
(3-myristoylaminopropyl)dimethylbenzy
lammonium chloride, m. 54°; this compound forms a clear 25% solution in H2O and is a germicide effective against Staphylococcus aureus in a dilution
of 1:25,000 at 37° in a 5-min. test and has a PhOH coefficient of 277-333; it is also a wetting agent for cotton fabrics. Using the same procedure, (3-lauroylaminopropyl)dimethylbenzylammonium chloride is obtained, which also is a good germicide.

ACCESSION NUMBER: 1949:23628 CAPLUS
DOCUMENT NUMBER: 43:23628
ORIGINAL REFERENCE NO.: 43:4430h-i,4431a-b
TITLE: Quaternary ammonium compounds
INVENTOR(S): Cook, Elmer W.; Moss, Philip H.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2459062		19490111	US	

L9 ANSWER 16 OF 16 CAPLUS COPYRIGHT 2005 ACS on STN
AB (Stearoylaminopropyl)dimethylbenzylammonium chloride was synthesized in 5 steps: (1) 25% aqueous Me2NH 545 g. was added to CH2:CHCN 170 g. below 20°, poured after 1 hr. into 350 cc. 10% aqueous NaOH, and the oily layer plus the ether extract dried and distilled to yield 218 g. of β-(dimethylamino)propionitrile, b22 73-4°. (2) Me2N(CH2)2CN 207 hydrogenated over **Raney Ni** at 100° and 90
atmospheric in the presence of NH3 72.4 yielded
3-(dimethylamino)propylamine,
b760 134°, 204.5 g. (3) C17H35COCl 49 was added dropwise to Me2N(CH2)3NH2 15.5 in C6H6 160 g. and the solution was washed after 1 hr. with 10% aqueous NaOH and H2O and distilled, giving a solid
N,N-dimethyl-3-
(stearoylamino)propylamine, b1-2 208-15°. (4) C17H35CONH(CH2)3NMe2 0.4 mol. was treated with C2H4O in the presence of 0.93 g. NaOH in tert-BuOH at 65°, and the NaOH neutralized with 1.9 cc. 38% HCl. (5) The product of step (4) was quaternized by reaction with 51 g.
PhCH2Cl
at 75° 2 hrs.; after filtering and evaporating off the solvent the quaternary amine salt was a crystalline solid at room temperature, soluble in aqueous Na2CO3
or H2O. Similar products are made using capryl, lauroyl or palmitoyl chloride in step (3) or 1-C10H7CH2Cl in step (5).

ACCESSION NUMBER: 1949:13222 CAPLUS
DOCUMENT NUMBER: 43:13222
ORIGINAL REFERENCE NO.: 43:2630i,2631a-c
TITLE: Aliphatic amide-substituted propyl quaternary ammonium
compounds
INVENTOR(S): Moss, Philip H.; Cook, Elmer W.
PATENT ASSIGNEE(S): American Cyanamid Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2459088		19490111	US	

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L1           302 S 1738-25-6/RN
L2          2673 S 109-55-7/RN
L3            44 S L1 AND L2
L4        787174 S NI OR NICKEL
L5            15 S L3 AND L4
L6         53134 S SPONGE OR RANEY
L7         27598 S L6 AND L4
L8            44 S L2 AND L1
L9            16 S L7 AND L1
L10           28 S L7 AND L2

=> s l10 not l9
L11           15 L10 NOT L9

=> s l11 not l5
L12           15 L11 NOT L5

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L12 ANSWER 1 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB Cationic surfactants R4CON(Z)(CH2)nNR1R2R3+ X- [R1-R3 = C1-4 alkyl, C2-4 hydroxyalkyl; R4 = C1-21 aliphatic hydrocarbyl; X = halogen, R3OSO3; Z = (glycosyl)alditol residue; n = 1-4] are prepared by quaternization of R4CON(Z)(CH2)nNR1R2 with R3X. Thus, reaction of glucose with H2N(CH2)3NMe2, followed by reduction with **Raney Ni**, gave N-[3-(dimethylamino)propyl]glucamine, which was acylated with CH2:CH(CH2)8COCl and quaternized with MeI to give the surfactant. Such surfactants are specifically used as hair conditioners, grinding aids, flocculating agents, and pigment dispersants.

ACCESSION NUMBER: 1999:111730 CAPLUS
DOCUMENT NUMBER: 130:169839
TITLE: Quaternary ammonium derivatives of amido alditols, their preparation, compositions containing them, and their uses
INVENTOR(S): Petit, Serge
PATENT ASSIGNEE(S): Ceca S. A., Fr.
SOURCE: Eur. Pat. Appl., 11 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 895979	A1	19990210	EP 1998-401601	19980626
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
FR 2767133	A1	19990212	FR 1997-10215	19970808
FR 2767133	B1	19990924		
JP 11106784	A2	19990420	JP 1998-223168	19980806
CA 2243503	AA	19990208	CA 1998-2243503	19980807
PRIORITY APPLN. INFO.:			FR 1997-10215 A	19970808

OTHER SOURCE(S): MARPAT 130:169839
REFERENCE COUNT: 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE
FORMAT

L12 ANSWER 2 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB R1R2NR3N[((CH2)3NH)mCOR4][(CH2)3NH]nCOR4 [I; R, R2 = C1-4 (hydroxy)alkyl; R3 = C2-6 alkylene, alkenylene; R4 = C7-35 alkyl, alkenyl; R1R2 may be combined with CR2, NR, or O to form a ring; R = H, C1-4 alkyl; m, n = 1-5], useful as surfactants and softening agents for fabric and hair (no data), are prepared by cyanoethylation of R1R2NR3NH2 with acrylonitrile, catalytic hydrogenation for aminopropylation, optional repeating the cyanoethylation and hydrogenation, then acylation of the resulting R1R2NR3N[((CH2)3NH)mH][(CH2)3NH]nH with R4CO2R5 (R5 = H, C1-3 alkyl). N,N-dimethylpropanediamine (102 g) was treated dropwise with 530 g acrylonitrile at 55-65° over 4 h, autoclaved in the presence of **raney Ni** at 70° and 20 kg/cm2-gage H for 10 h to give 125 g amines, which (100 g) was treated with 240 g octadecanoic acid at 180° for 8 h to give 256 g I (R1 = R2 = Me, R3 = CH2CH2, R4 = C17H35, m = n = 1).

ACCESSION NUMBER: 1994:298091 CAPLUS
DOCUMENT NUMBER: 120:298091
TITLE: Preparation of diamidoamines as surfactants and softening agents for fabric and hair
INVENTOR(S): Tomifuji, Takeshi; Kato, Tooru; Sotodani, Koshiro
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06016608	A2	19940125	JP 1992-175265	19920702
PRIORITY APPLN. INFO.:			JP 1992-175265	19920702

OTHER SOURCE(S): MARPAT 120:298091

L12 ANSWER 3 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

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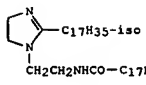

IV

AB The claimed title compds., for use in pharmaceuticals and cosmetics (no data), are derivs. of isostearic acid (I) which are analogous to derivs. of other acids (e.g., lauric, myristic, cetylic, stearic, oleic, and ricinoleic acids), and are elaborated from the following compds.: isostearyldimethylamine (II), (isostearylamidopropyl)dimethylamine [i.e. iso-C17H35CONH(CH2)3NMe2] (III), and diisostearylimidazoline (sic, i.e. IV). Thus, conversion of I to its nitrile and reduction of this with **Raney Ni** gave 87% isostearylamine, which can be converted to II by reductive methylation. Alternatively, hydrogenation of isostearyl alc. and Me2NH with a Cr salt catalyst gave 93.3% II. Amidation of I with H2N(CH2)3NMe2 gave 85% III, whereas reaction of I with diethylenetriamine gave IV. Quaternization of III with MeCl or PhCH2Cl gave corresponding quaternary ammonium chlorides in 90% and 98% yields, resp. Alternatively, quaternization of III with ClCH2CO2Na or Cl(CH2)3SO3Na gave corresponding betaines [e.g., iso-C17H35CONH(CH2)3N+Me2CH2CO2-], whereas oxidation of III with peroxide gave the N-oxide.

ACCESSION NUMBER: 1994:191144 CAPLUS
DOCUMENT NUMBER: 120:191144
TITLE: Nitrogen containing compounds derived from isostearic acid
INVENTOR(S): Costabile, Jose Antonio
PATENT ASSIGNEE(S): Quimica Nacional Quiminasa S/A, Brazil
SOURCE: Braz. Pedido PI, 10 pp.
CODEN: BPXXDX
DOCUMENT TYPE: Patent
LANGUAGE: Portuguese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
BR 9300059	A	19930824	BR 1993-59	19930108
PRIORITY APPLN. INFO.:			BR 1993-59	19930108

L12 ANSWER 4 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN

AB R1R2NR3N(CmH2mOCOR4)[CH2CH2CH2NH]nCOR4 [I; R1, R2 = C1-4 (hydroxy)alkyl; R1R2 may form ring linked via CR2, NR, or O; R = H, C1-4 alkyl; R3 = C2-6 alkylene, alkenylene; R4 = C7-35 alkyl, alkenyl; n = 1-3; m = 2-9], useful as surfactants or softeners for textiles, hair, etc. (no data), are prepared by hydroxyalkylation of R1R2NR3NH2 (R1, R2 = same as I), cyanoethylation-hydrogenation of R1R2NR3NHCmH2mOH (R1-3, m = same as I), optional repeating of the cyanoethylation and hydrogenation, and acylation of the resulting R1R2NR3N(CmH2mOH)[CH2CH2CH2NH]nH (R1-3, m, n = same as I) with R4CO2R5 (R4 = same as I; R5 = H, C1-3 alkyl). N,N-dimethylaminopropanediamine (309 g) was treated with 111 g ethylene oxide at 150-170° for 1 h to give 122 g amino alc., which (100 g) was mixed with acrylonitrile at 55-65° over 4 h and hydrogenated with **Raney Ni** under 20 kg/cm2-G H at 70° for 8 h to give 86 g amine. Acylation of 64 g the amine with 166 g octadecanoic acid at 180° for 18 h gave 206 g I (R1 = R2 = Me, R3 = CH2CH2CH2, R4 = C17H35, m = 2, n = 1).

ACCESSION NUMBER: 1994:163477 CAPLUS
DOCUMENT NUMBER: 120:163477
TITLE: Amines as surfactants or softeners and their preparation
INVENTOR(S): Fudo, Takeshi; Kato, Tooru; Sotodani, Koshiro
PATENT ASSIGNEE(S): Kao Corp, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05246965	A2	19930924	JP 1992-45412	19920303
PRIORITY APPLN. INFO.:			JP 1992-45412	19920303

OTHER SOURCE(S): MARPAT 120:163477

L12 ANSWER 5 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
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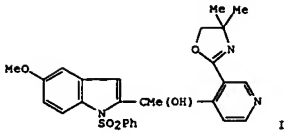

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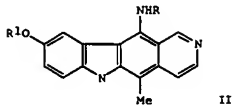

II

AB A route to 11-amino-substituted 6H-pyrido[4,3-b]carbazoles was developed. Thus, condensation of 2-(4-lithiopyrid-3-yl)-4,4-dimethyloxazoline with 2-acetyl-5-methoxy-1-phenylsulfonylindole led to a low yield of the expected alc. I, which upon hydrolysis gave a complex mixture A better starting building block was 4-acetyl-N,N-diisopropylnicotinamide obtained either from N,N-diisopropyl-4-lithionicotinamide (low yield) or from pyridine-3,4-dicarboxylic anhydride, using a 4-step sequence. This compound was treated with 2-lithio-5-methoxy-1-phenylsulfonylindole, affording N,N-diisopropyl-4-[1-(5-methoxy-1-phenylsulfonylindol-2-yl)-1-hydroxymethyl]nicotinamide. Hydrolysis and then reduction led to 4-[1-(5-methoxy-1-phenylsuylfonylindol-2-yl)-ethyl]nicotinic acid whose amides were cyclized by phosphorus trichlorideoxide. Finally, the title compds. II (R = CH2CH2NMe2, R1 = Me, H; R = CH2CH2CH2NR2, R1 = Me, R2 = Me, Et) were obtained by **Raney nickel** reduction and elimination of the 6-phenylsulfonyl protecting group. A screen of II for cytotoxicity and neoplasm inhibiting activity is also reported.

ACCESSION NUMBER: 1992:128707 CAPLUS
DOCUMENT NUMBER: 116:128707
TITLE: Synthesis of 11-amino-substituted 9-methoxy-5-methyl-6H-pyrido[4,3-b]carbazoles
AUTHOR(S): Praly-Deprez, Isabelle; Rivalle, Christian; Huel, Christiane; Belehradek, Jean; Paoletti, Claude; Bisagni, Emile
CORPORATE SOURCE: Sect. Biol., Inst. Curie, Orsay, 91405, Fr.
SOURCE: Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999) (1991), (12), 3165-71
CODEN: JCPRB4; ISSN: 0300-922X
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 6 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB Long chain alkyl amines, suitable for use as lubricant oil or gasoline additives, are prepared by (1) reaction of a polyolefin having a mol. weight 330-2000 with O3 in the presence of a solvent; (2) reaction of the ozonolysis product without separation and/or isolation of the carbonyl compds. formed with primary hydrocarbyl amines to form an imine, (3) hydrogenating the resulting imine in the presence of a hydrogenation catalyst, and (4) recovering the long chain alkyl amine from the hydrogenation products. Thus, O3 was passed through a mixture of 100 g polyisobutene and 100 mL n-hexane for 4 h at 0.13 mol/h and 15° and after passing briefly N through the reaction mixture to remove unreacted O3 0.2 mol dimethylaminopropylamine was added. Then, the mixture was refluxed 2 h at 70° and evaporated under vacuum at 140° to give 115 g an imine which (70 g) was autoclaved with 5 g **Raney Ni** in EtOH-cyclohexane under 15MPa H at 90° for 19 h to give a straw colored residue having no imine or carbonyl functions.

ACCESSION NUMBER: 1991:81022 CAPLUS
DOCUMENT NUMBER: 114:81022
TITLE: Synthesis of hydrocarbyl amines from polyolefins
INVENTOR(S): Blackborrow, John Richard; Peretti, Regis
PATENT ASSIGNEE(S): BP Chimie S. A., Fr.
SOURCE: Eur. Pat. Appl., 5 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 384086	A1	19900829	EP 1989-400383	19890210
R: FR				
WO 9009371	A1	19900823	WO 1990-GB144	19900201
W: AU, BR, CA, HU, JP, NO, SU, US				
RW: AT, BE, CH, DE, DK, ES, FR, GB, IT, LU, NL, SE				
AU 9050286	A1	19900905	AU 1990-50286	19900201
AU 622286	B2	19920402		
EP 411084	A1	19910206	EP 1990-902664	19900201
EP 411084	B1	19931013		
R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE				
BR 9005079	A	19910806	BR 1990-5079	19900201
JP 03504505	T2	19911003	JP 1990-502996	19900201
HU 57702	A2	19911230	HU 1990-1718	19900201
HU 208666	B	19931228		
AT 95809	E	19931015	AT 1990-902664	19900201
ES 2045907	T3	19940116	ES 1990-902664	19900201
ZA 9000872	A	19911030	ZA 1990-872	19900206
CA 2009557	AA	19900810	CA 1990-2009557	19900208
IN 177255	A	19961214	IN 1990-DE115	19900209
US 5103061	A	19920407	US 1990-571652	19900906
NO 9004210	A	19900927	NO 1990-4210	19900927
NO 171058	B	19921012		
NO 171058	C	19930120		

PRIORITY APPLN. INFO.: EP 1989-400383 A 19890210
FR 1989-40038 A 19890210

L12 ANSWER 6 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

EP 1990-902664	A	19900201
WO 1990-GB144	A	19900201

L12 ANSWER 7 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
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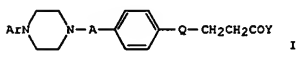

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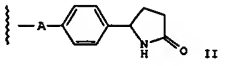

II

AB The title compds. [I; Ar = (hetero)aryl; A = alkylene; Q = CH(NH2), C:NOH, CH(OH), CO, CH2; Y = OR, NR1R2; R = H, alkyl; R1, R2 = H, alkyl, aralkyl, BNR3R4; or NR1R2 = 5- to 7-membered N-containing heterocyclyl; R3, R4 = H, alkyl, aralkyl; B = alkylene] or their pharmaceutically acceptable acid addition salts are prepared I have antihistaminic and coronary blood flow-increasing activity and are useful as allergy inhibitors and pharmaceuticals for treating heart diseases (no data) as well as intermediates for psychotropics (II) such as antipsychotics, antidepressants, or anxiolytics. Thus, Et 4-oxo-4-[4-(4-chlorobutyl)phenyl]butyrate and 1-(3-methylphenyl)piperazine were dissolved in a mixture of DMF and PhMe and after adding K2CO3, the resulting mixture was refluxed 18 h to give I [Ar = 3-MeC6H4, A = (CH2)4, Q = CO, Y = OEt]. Saponification of the latter followed by oximation with NH2OH.HCl in refluxing EtOH in the presence of NaHCO3 gave I [Ar, A = same as above, Q = C:NOH, Y = OH] which was hydrogenated over **Raney Ni** in MeOH to give II (Ar, A = same as above).

ACCESSION NUMBER: 1989:632864 CAPLUS
DOCUMENT NUMBER: 111:232864
TITLE: Preparation of 4-[4-[(4-arylpiperazin-1-yl)alkyl]phenyl]butyric acid derivatives as drugs or intermediates for psychotropics
INVENTOR(S): Nakao, Tatsu; Morita, Kenji; Obata, Minoru; Morimoto, Yasuto
PATENT ASSIGNEE(S): Yoshitomi Pharmaceutical Industries, Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 14 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 01113377	A2	19890502	JP 1987-268748	19871023

PRIORITY APPLN. INFO.: JP 1987-268748 19871023

OTHER SOURCE(S): MARPAT 111:232864

L12 ANSWER 8 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
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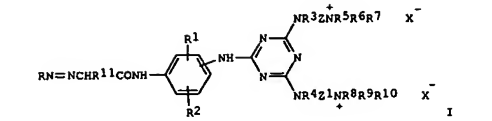

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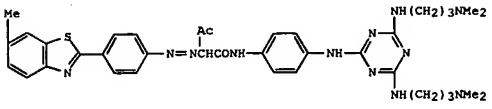


II

AB Fast yellow dyes of general structure I are described, where R represents the radical of an anionic group-free diazo component; R1 and R2 are H and(or) nonionic substituents; R3 and R4 are H, C1-4 alkyl, or C1-4 hydroxyalkyl; R5-R10 are H, C1-4 alkyl, Ph, etc.; R11 is Ac, CN, CO2Me, etc.; Z and Z1 are bridging groups or single bonds; and X- is an anion.
I
are dyes for cellulosic materials, especially paper. Thus, reaction of cyanuric
chloride [108-77-0] with p-O2NC6H4NH2 [100-01-6] and then Me2N(CH2)3NH2 [109-55-7], reduction over **Raney Ni**, treatment
with diketene, and coupling with diazotized dehydrothio-p-toluidine [92-36-4] gave a dye (II) [91458-37-6] which, when dissolved in HOAc, formed a stable solution for dyeing cellulosic materials in greenish yellow
shades. Quaternization of II with Me2SO4 followed by dissoln. in HOAc also gave a solution for producing greenish yellow dyeings.

ACCESSION NUMBER: 1984:492780 CAPLUS
DOCUMENT NUMBER: 101:92780
TITLE: Cationic triazine dyes
INVENTOR(S): Kunde, Klaus
PATENT ASSIGNEE(S): Bayer A.-G. , Fed. Rep. Ger.
SOURCE: Ger. Offen., 33 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

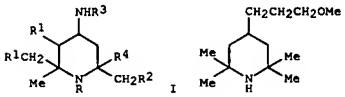
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3048998	A1	19820715	DE 1980-3048998	19801224
EP 57245	A2	19820811	EP 1981-106548	19810824
EP 57245	A3	19820901		

L12 ANSWER 8 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

EP 57245	B1	19840530		
R: CH, DE, FR, GB, IT				
JP 57131257	A2	19820814	JP 1981-205385	19811221
JP 02003825	B4	19900125		

PRIORITY APPLN. INFO.: DE 1980-3048998 A 19801224

L12 ANSWER 9 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
GI



I

II

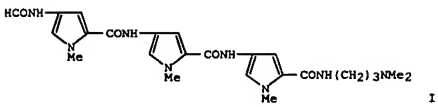
AB Title piperidines I [R = H, C1-12 alkyl; R1, R2 = H, C1-5 alkyl, R4 = Me; R2R4 = 2,2,6,6-tetramethylpiperidin-4,4-diyl; R3 = alkoxy-, hydroxy- or amino-substituted C1-18 alkylene, alkyleneoxy, C5-12 cycloalkylene or poly(aminoalkylene)] were prepared by reductive amination of the corresponding 4-piperidinones. Thus, hydrogenating 155 g 2,2,6,6-tetramethyl-4-piperidinone with 178 g MeO(CH2)3NH2 over **Raney Ni** moistened with MeOH 4 h at 80°/20 bar gave 91% II.

ACCESSION NUMBER: 1981:619971 CAPLUS
DOCUMENT NUMBER: 95:219971
TITLE: Polyalkylpiperidylamines
INVENTOR(S): Wiezer, Hartmut
PATENT ASSIGNEE(S): Hoechst A.-G. , Fed. Rep. Ger.
SOURCE: Ger. Offen., 17 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3007996	A1	19810917	DE 1980-3007996	19800301

PRIORITY APPLN. INFO.: DE 1980-3007996 A 19800301

L12 ANSWER 10 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
GI

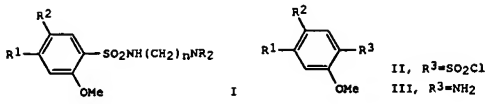


I

AB The title compound I was prepared from 4-nitro-1-methyl-2-pyrrolecarboxylic
acid (III) with H2NCH2CH2CH2NMe2 to give the amide, which was reduced in the presence of **Raney Ni** to give an amine which was successively treated with the acid chloride of II, reduced, treated with the acid chloride of II, reduced and formylated.

ACCESSION NUMBER: 1978:50585 CAPLUS
DOCUMENT NUMBER: 88:50585
TITLE: Analog of Distamycin A with a dimethylamino group
AUTHOR(S): Glibin, E. N.; Tsukerman, B. V.; Ginzburg, O. F.
CORPORATE SOURCE: Leningr. Tekhnol. Inst., Leningrad, USSR
SOURCE: Zhurnal Organicheskoi Khimii (1977), 13(10), 2231-2
CODEN: ZORKAE; ISSN: 0514-7492
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L12 ANSWER 11 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
GI



AB Benzenesulfonamides I [R = Me, Et, R2 = (CH2)2O(CH2)2, (CH2)4, (CH2)5, R1 = R2 = H; R1 = NO2, R2 = H, Cl; R1 = H, R2 = Cl, n = 2, 3; R = Me, Et, R2 = (CH2)2O(CH2)2, (CH2)5, R1 = H, R2 = SO2NH2] (36 compds.), useful as antiemetics, local anesthetics, anticonvulsants, and bacteriostatics, were
prepared by treating sulfonyl chlorides II with amines H2N(CH2)nNR2. Hydrogenation of the nitro compds. over **Raney Ni** gave the corresponding amines I (R1 = NH2) (16 compds.). II were prepared by known methods from III (R1 = R2 = H; R1 = NO2, R2 = Cl), 4-ClC6H4R4 (R4 = OH, OMe), or 2-O2NC6H4OMe. The antiemetic activities for selected I were tabulated.

ACCESSION NUMBER: 1977:171084 CAPLUS
DOCUMENT NUMBER: 86:171084
TITLE: N-Substituted 2-methoxybenzenesulfonamides
INVENTOR(S): Moreau, Robert C.; Fournier, Jean P.
PATENT ASSIGNEE(S): Choay, S. A., Fr.
SOURCE: Ger. Offen., 36 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2623447	A1	19770113	DE 1976-2623447	19760525
FR 2313918	A1	19770107	FR 1975-17973	19750609
FR 2313918	B1	19781006		
AT 7604019	A	19800215	AT 1976-4019	19760601
AT 358555	B	19800925		
CA 1083573	A1	19800812	CA 1976-253973	19760603
JP 52031044	A2	19770309	JP 1976-65723	19760607
JP 60049630	B4	19851102		
DK 7602523	A	19761210	DK 1976-2523	19760608
DK 146592	B	19831114		
DK 146592	C	19840612		
SE 7606440	A	19761210	SE 1976-6440	19760608
SE 430248	B	19831031		
SE 430248	C	19840209		
NL 7606172	A	19761213	NL 1976-6172	19760608
ES 448646	A1	19770701	ES 1976-448646	19760608
US 4132786	A	19790102	US 1976-693896	19760608
GB 1545628	A	19790510	GB 1976-23701	19760608
CH 616917	A	19800430	CH 1976-7179	19760608
BE 842753	A1	19761209	BE 1976-167761	19760609
US 4211776	A	19800708	US 1978-947623	19781002

L12 ANSWER 11 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

AT 7901500	A	19800615	AT 1979-1500	19790227
AT 360505	B	19810112		
JP 60243060	A2	19851203	JP 1985-91304	19850430

PRIORITY APPLN. INFO.: FR 1975-17973 A 19750609
AT 1976-4019 A 19760601
US 1976-693896 A3 19760608

L12 ANSWER 12 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB Incorporation of N-(polyhydroxyalkyl)amines, prepared by catalytic reductive
amination of monosaccharides or uronic acids, into skin care prepns. promoted water retention by the skin, thus maintaining its softness and elasticity. For example, N-(2,3-dihydroxypropyl)glucamine [57273-24-2] was prepared by catalytic hydrogenation of the reaction product of 19.6 g D-glucose [50-99-7] and 18.2 g 2,3-dihydroxypropylamine [616-30-8] in H2O-MeOH at 50-70° and 180 atm with **Raney Ni**. A baby cream was prepared from e.g. N-(β-piperazinoethyl)glucamine lactate [57288-68-3] 5.0, Dehymuls E 7.0, decyl oleate 10.0, vaseline 10.0, wool fat 5.0, boric acid 0.2, talcum 12.0, ZnO 8.0, Nipagin M 0.2, and water 42.6 parts by weight

ACCESSION NUMBER: 1976:8854 CAPLUS
DOCUMENT NUMBER: 84:8854
TITLE: Skin-treating and skin-protective composition
INVENTOR(S): Moeller, Hinrich; Osberghaus, Rainer; Gloxhuber, Christian; Braig, Siegfried
PATENT ASSIGNEE(S): Henkel und Cie. G.m.b.H., Fed. Rep. Ger.
SOURCE: Ger. Offen., 25 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2404070	A1	19750814	DE 1974-2404070	19740129
SE 7500075	A	19750730	SE 1975-75	19750103
NL 7500071	A	19750731	NL 1975-71	19750103
FR 2258834	A1	19750822	FR 1975-2610	19750128
FR 2258834	B1	19781103		
JP 50111241	A2	19750901	JP 1975-11047	19750128
AT 7500627	A	19760715	AT 1975-627	19750128
AT 335621	B	19770325		
US 4021539	A	19770503	US 1975-544859	19750128
GB 1497875	A	19780112	GB 1975-3569	19750128
BE 824914	A1	19750729	BE 1975-152820	19750129
CH 609240	A	19790228	CH 1975-1023	19750129

PRIORITY APPLN. INFO.: DE 1974-2404070 A 19740129

L12 ANSWER 13 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB Glucose reacted with RRN(CH2)3NH2 (R = Me, Et, CH2CH2OH) and EtOH containing
Raney Ni in an autoclave followed by treatment with R1NCO [R1 = Me(CH2)n, n = 11, 13, 15,17] to give R1NHCON[CH2[CH(OH)]4CH2OH](CH2)3NRR, which showed a fluorescent whitening activity on wool, polypropylene, polyester, and polyurethane textiles.

ACCESSION NUMBER: 1975:497843 CAPLUS
DOCUMENT NUMBER: 83:97843
TITLE: N-Alkyl-N'-polyhydroxyalkyl-N'-aminoalkyl ureas and their use in washing compositions
INVENTOR(S): Eckert, Hans W.
PATENT ASSIGNEE(S): Henkel und Cie. G.m.b.H., Fed. Rep. Ger.
SOURCE: Ger. Offen., 18 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2349278	A1	19750410	DE 1973-2349278	19731001

PRIORITY APPLN. INFO.: DE 1973-2349278 A 19731001

L12 ANSWER 14 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB The use of hydroxides of alkali or alkaline earth metals, metal alcoholates, or amides in the hydrogenation of nitriles was described. To a mixture of 100 g. adiponitrile and 15 g. MeOH are added 1.5 g. powdered KOH and 40 g. Raney Ni. The mixture is heated in an autoclave in a H stream (150 kg./cm.2 pressure) at 30-2° 1 hr. and distilled to give 102.5 g. hexamethylenediamine, b. 195-205°. Manufacture of bis(3-aminopropyl)-methylamine (b2 97-100°), 3-(dimethylamino)propylamine, and 2-(diethylaminoethyl) 3-aminopropyl ether from bis(2-cyano-ethyl)methylamine, 2-(dimethylamino)propionitrile, and 2-diethylaminoethyl 2-cyanoethyl ether, resp., was also described.

ACCESSION NUMBER: 1964:15924 CAPLUS
DOCUMENT NUMBER: 60:15924
ORIGINAL REFERENCE NO.: 60:2756f-g
TITLE: Amines from nitriles
INVENTOR(S): Taniyama, Masakazu; Sawa, Natsuo; Nagaoka, Takeshi; Takada, Toshihiro
PATENT ASSIGNEE(S): Toho Rayon Co., Ltd.
SOURCE: 2 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 38021353		19631014	JP	19580227

L12 ANSWER 15 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN
AB Mixed anhydrides with mono-Et carbonate are particularly useful intermediates for the synthesis of the N-(ω-dialkylaminoalkyl)amides of 3,4,5-(MeO)3C6H2CO2H (I) and of the 2-Br derivative (II) of I. Me ester (III) (1.25 g.), m. 68-72°, of I refluxed 4 hrs. with 5.98 g. Me2N(CH2)3NH2 and 8 cc. MeOH at 125-30°, and the mixture kept at 130°/16 mm. left only III. The 4-OH analog of III, m. 103-6°, behaved similarly. I (42.4 g.) in 600 cc. C6H6 treated with 20.2 g. Et3N, cooled, treated with 22-3 g. ClCO2Et (IV), kept 0.5 hr. at room temperature, treated with 0.2 mole of the appropriate dialkylaminoalkylamine (V), and filtered after 15 hrs., the residue washed with C6H6, the combined filtrates evaporated, the residual oil extracted with dilute HCl, the acid extract basified with NaOH, and the crude precipitate digested with Et2O and recrystd. (ligroine) gave the corresponding 3,4,5-(MeO)3C6H2CONH(CH2)nNR2 (R, n, g. V used, g. recovered I, g. yield and m.p. of product given): Et, 3 (VI), 26.0, 14.2, 46.4, 59.61°; Me, 3 (VII), 20.5, 12.7, 41.0, 83-5°; Et, 2 (VIII), 26.0, 12.8, 42.6, 105-7°; Me, 2 (IX), 9.2, 7.8, 18.7, 119-20°. Bu3N salt of 0.1 mole I (from 21.2 g. I and 18.5 g. Bu3N) in 300 cc. C6H6 treated with 15 g. IV, the mixture treated after 1 hr. with NH3, and the precipitate filtered off gave 13.74 g. amide of I, m. 169-71° (PhNO2). II (100 g.), 36.2 g. Et3N, and 38.5 g. IV brought to reaction in 500 cc. CHCl3, 1/2 of the solution treated with gaseous NH3, kept 1 hr., and evaporated, and the residue extracted with NH4OH to remove 5.51 g. unreacted II and recrystd. (H2O) yielded 25.11 g. amide of II, needles, m. 166-8°. A slight excess of the appropriate V added to 1/8 of the original mixed anhydride solution, the mixture processed in the usual manner, and the crude products recrystd. (hot ligroine) yielded the corresponding 2,3,4,5-Br(MeO)3C6HCONH(CH2)nNR2, the 2-Br derivs. of the following compds. (g. V used, g. II recovered, % crude and pure yield, and m.p. of product given): VI, 5.78, -, 14.44, 10.18, 65-7°; VII, 4.82, -, 17.05, -, 87-9°; VIII, 5.19, 4.71, 12.92, 11.00, 37-50°; IX, 3.80, 4.50, -, 8.34, 51-7°. Bu3N.BzOH (0.1 mole) (from 12.2 g. BzOH and 18.5 g. Bu3N) in 150 cc. C6H6 treated with 10.9 g. IV, the solution treated after 2 hrs. with 10.0 g. PhNH2 and allowed to stand, and the deposit filtered off gave 14.1 g. BzNHPh, m. 155-7°; an addnl. 0.88 g. was isolated from the C6H6 phase; the aqueous alkaline washings of the C6H6 phase acidified yielded 2.8 g. BzOH. p-MeC6H4SO2Cl (19.05 g.) in 50 cc. CHCl3 solution added to 0.1 mole Et3N.BzOH in 80 cc. CHCl3, the mixture treated with dry NH3, the CHCl3 evaporated, and the residue treated with H2O left 2.77 g. BzNH2, m. 124-6°. Me2N(CH2)2Cl.HCl (14.4 g.) and 13.0 g. NaN3 in 55 cc. H2O heated 8 hrs. on the H2O bath, kept 15 hrs. at room temperature, treated with 4 g. NaOH in 15 cc. H2O, extracted after 2.5 hrs. 7 hrs. with Et2O, and the extract worked up gave 5.04 g. Me2N(CH2)2N3 (X), b16 32-40°; picrate, deep yellow needles, m. 114-15°. X (31 g.)

L12 ANSWER 15 OF 15 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
in 100 cc. H2O and 50 cc. MeOH hydrogenated over Raney Ni at 170 atm., filtered, acidified with concd. HCl, and evapd. gave 38 g. Me2N(CH2)3NH2.2HCl (XI.2HCl), which heated with 40 g. CaO at 150-95° yielded 18.34 g. XI, b. 103°; 3,5-(O2N)2C6H3CONH(CH2)3NMe2, m. 66-7° (aq. MeOH); XI.2HCl, m. 183°. Et2N(CH2)2Cl.HCl (17.2 g.), 10.0 g. KCN, 3.0 g. NaI, and 10 cc. H2O heated 6 hrs. in an autoclave at 140-70°, treated with 10 g. NaOH, and extd. 1.5 hrs. with Et2O, and the ext. worked up yielded 6.0 g. brown, fuming amine; a 3.98-g. portion distd. gave 3.26 g. Et2N(CH2)2CN (XII), b16 62-77°, n20D 1.4378, d24 0.8165; XII.EtI, m. 224-7° (decompn.) (MeOH). A similar run in an open vessel at reflux yielded 30% XII, MRD 38.16. XII (12.68 g.) in 55 cc. Et2O treated dropwise with 0.1 mole LiAlH4 in 140 cc. Et2O with stirring, refluxed 6 hrs., and worked up in the usual manner yielded 7.52 g. Et3N(CH2)3NH2, n20D 1.4436, d20 0.8306.

ACCESSION NUMBER: 1959:83235 CAPLUS
DOCUMENT NUMBER: 53:83235
ORIGINAL REFERENCE NO.: 53:14992i,14993a-h
TITLE: Trimethoxyphenyl derivatives. I. Synthesis of aminoamines via mixed anhydrides
AUTHOR(S): Schiemenz, Gunter Paulus; Engelhard, Hermann
CORPORATE SOURCE: Univ. Gottingen, Germany
SOURCE: Chemische Berichte (1959), 92, 857-62
CODEN: CHBEAM; ISSN: 0009-2940
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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